A STRUCTURE - REACTIVITY STUDY OF SELECTED PHOSPHORUS COMPOUNDS

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by

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This work is dedicated to my wife, Diana.

CONTENTS

ABSTRACT

The X-ray crystal structure determinations of a series of ring substituted dimethyl-N-phenyl-phosphoramidates, (R)PhNHP$(OCH_3)_2$, (R = *p*-OMe, H, *p*-NO_2, *o*-Et and 2,6-diMe) have confirmed the preference of the phosphoramidates for dimer formation which was proposed earlier from infrared data. The formation of N – H ---- O = P hydrogen bonded dimeric species is sensitive to steric effects and the presence of bulky substituents at the *ortho* positions of the benzene ring prevents dimer formation.

The X-ray crystal structure determination of the carboxylic analogues of the phosphoric amides above, (R)PhNHC(O)R' (R = *p*-OMe, *p*-NO_2 and R' = Me or Ph), serves to indicate the differences in the bonding between the phosphoric and carboxylic amides. The geometry of the amide group in the carboxylic amides is subject to the influence of intermolecular hydrogen bonding effects while the phosphoric amide group in the dimer is less sensitive to other intermolecular interactions. In the phosphoric amides the distortion of the amide group from the favourable (planar) geometry arizes principally from intramolecular steric repulsions.

The bond lengths determined for the phosphoric amides indicate that the phosphoric amide group is less conjugated than the corresponding carboxylic amide group. The P = O bond order is insensitive to changes in substituent electron-releasing or -withdrawing ability. The solid state data indicate that the electron density on the nitrogen

atom is not transferred to the phosphoryl group as readily as electron density is transferred from the nitrogen atom to the carbonyl group in carboxylic amides. This had previously been suggested from other spectroscopic studies on phosphoric and carboxylic amides. Therefore the approximate reciprocal dependence between the C = O and C – N bond lengths observed in carboxylic amides does not occur in the phosphoric amides. The changes in the electron-withdrawing or -releasing properties of the ring substituent in the phosphoric amides is confined to changes in the acidity of the amide hydrogen atom and is correlated with the N – H ---- O = P hydrogen bond length.

The angular dependence of the approach of nucleophiles to quaternary phosphonium ions L_4P^+ was investigated. The analysis of 36 structures of the general formula $L_4P^+X^-$ indicates that the nucleophile generally adopts the "face" approach to the phosphonium centre. When the expected leaving group on the phosphorus atom has an acidic α-proton the nucleophile approaches adjacent to the leaving group as a result of a strong interaction between the α-proton and the nucleophile. This tendency had been found previously and is confirmed by this study. When the expected leaving group is of the form OR or NR and there is no possibility of interaction between the nucleophile and the leaving group the nucleophile approaches the phosphonium centre opposite the leaving group. The angular dependence of the approach of the nucleophiles to the phosphonium centres appears to be as a result of packing factors.

The crystal structures of the hydrated and dehydrated forms of synthetic analcime indicate that the structures are essentially identical to structure of the natural form determined previously.

CHAPTER ONE

1. GENERAL INTRODUCTION

1.1 Recent Developments in Chemical Crystallography

In recent years advances in instrumentation and computing power have made the determination of crystal structures easier and faster. Unfortunately, while progress in the determination of structures has been rapid, the ability to describe and hence understand these structures has progressed more slowly. In the past, analysis of a crystal structure was often limited to a discussion of bond lengths, angles, conformation and packing. It is now however being realized that much more chemical information is available from the detailed analysis of the relationships between these solid state parameters; information that was often overlooked in the past. Of particular importance is the role of molecular interactions and non-bonded distances. These studies are revealing new information on secondary bonding, structural correlations (reaction pathways) and reaction mechanisms, to name only a few.[1,2]

The Cambridge Crystallographic Data Centre (CCDC) maintains a computer data file which stores information on crystal structure determinations by X-ray or neutron diffraction of compounds containing at least one carbon atom.[3,4] This file is updated regularly and is available throughout the world via affiliated centres. Chemists thus have an immense source of structural data readily available. Using the CCDC file, chemists are now analysing in a complete way previously determined structures, or, more usefully, series of related structures.[5] In cases where chemically useful structural data are not available, structures will be determined.

1.2 Structure-Reactivity Relationships

A particularly active field of interest is that of structure-reactivity relationships. Correlation is sought between the geometrical parameters (i.e. solid state parameters) and the properties of the compound or series of compounds in solution.[6] In this way correlations between, for example, bond length and reactivity[7] and bond length and ^1H n.m.r. data have been undertaken.[8] This information has yielded new insights into the nature of bonding in the compounds studied.

Access to the CCDC data file permits the derivation of information about reaction paths by searching for certain kinds of correlations among the structural parameters for a group of related molecules or crystals. This is known as the "Structural Correlation Principle".[9] To quote from Dunitz[9]: "The idea is to select some structural fragment or subunit that occurs in a wide variety of environments and try to correlate changes that occur in one structural parameter with changes that occur in others. If correlations between these parameters can be found, the various copies of the fragment can be ordered in a sequence that can be interpreted in terms of a gradual deformation of the fragment, just as a series of 'still' pictures, arranged in the right order, can be viewed cinematically." Application of the "Structural Correlation Principle" to a number of systems has yielded remarkable results which shed light on the deformations undergone by molecules passing through chemical reaction pathways.

1.3 Aims of this Research Project

The research project is divided into two parts; the first part involves

the determination of the crystal structures of a number of phosphoric and carboxylic amides so that the geometrical parameters can be correlated with previously determined spectroscopic data. It is hoped that this will shed further light on the nature of the bonding in these compounds. The second part involves the application of the "Structural Correlation Principle" to phosphonium salts.

CHAPTER TWO

2. GENERAL EXPERIMENTAL PROCEDURES USED IN THE CRYSTALLOGRAPHIC ANALYSIS

2.1 Introduction

This chapter outlines the experimental procedures which are common to all the compounds which have been studied by the methods of single crystal X-ray crystallography.

2.2 Single Crystal Analysis[10,11]

Preliminary crystallographic information was obtained from oscillation, Weissenberg and precession photography. X-radiation (nickel-filtered CuK_α, λ = 1.5418 Å) for the photographic work was generated by Philips X-ray generators (PW 1120 and PW 1140) operating at 0.8 kW. The choice of space group was made on the basis of the photographic results and confirmed by diffractometer data.

Suitable single crystals of each compound were mounted on glass fibres and sent to the National Physical Research Laboratory (N.P.R.L.) at the Council for Scientific and Industrial Research (C.S.I.R.), Pretoria, for diffractometer data collection. The diffractometer data collection was carried out on a Philips PW 1100 computer-controlled four circle diffractometer operating in the ω - 2θ scan mode. In all cases graphite monochromated MoK_α, λ = 0.7107 Å, X-radiation was generated by a Philips 1140 generator operating at 20 mA and 50 kV. All data collections were carried out at room temperature. Lorentz-polarization corrections were applied to all data immediately after data collection. The data were returned and stored on magnetic tape. Absorbtion corrections

were not applied to the data.

Accurate lattice constants were obtained by a least-squares fit of the χ, ϕ and 2θ angles of 25 reflections accurately centred on the diffractometer. Crystal and instrumental stability were monitored by measuring the intensities of three reference reflections periodically. The standard error (σI_{rel}) in the relative standard intensity, I_{rel}, was calculated from the formula:

$$\sigma(I_{rel}) = [N_{pk} + N_{bg} + N_{instr}]^{\frac{1}{2}}$$

where N_{pk} = gross peak count for the reflection

N_{bg} = background count as measured on either side of the peak

N_{instr} = $(0.02\ (N_{pk} - N_{bg}))^2$

Complete details of the equipment used in the X-ray analysis and diffractometer data collection will be summarized in Table 2.1.

2.3 Computation

Most calculations were carried out on a UNIVAC 1100 computer system at the University of Cape Town. The program SHELX[12] was employed for the solution and refinement of the structures. The program XANADU[13] was used to calculate geometrical parameters. In addition the subroutine BONDLA of the X-RAY 76[14] program system was used to determine estimated standard deviations in certain geometrical parameters. Molecular illustrations and projections were produced by the programs PLUTO[15] and PLUTOX.[16] An APPLE II micro-computer was also employed in certain routine calculations (refer to Appendix 3). Most of the tables in

this thesis were produced by the PRINTIT[17] word processing package maintained by the University of Cape Town Computer Centre.

Table 2.1 Details of Equipment used in X-ray Analysis

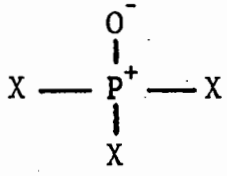
Single Crystal X-ray Photography:	
Radiation	CuK_α (λ = 1.5418Å)
Camera type	Stoe (Weissenberg & Precession)
Camera radius	28.65 mm
X-ray generators	Philips (PW 1120, PW 1140)
Operating conditions	40 kV, 20 mA
X-ray film	3M medical film
Single Crystral Diffractometry:	
Radiation	MoK_α (λ = 0.7107 Å)
Diffractometer (4-circle)	Philips PW 1100
X-ray generator	Philips PW 1140
Operating conditions	50 kV, 20 mA
Scan mode	ω - 2θ
Operating temperature	20 - 25°C
Location	N.P.R.L., C.S.I.R.

PART A : CHAPTER THREE

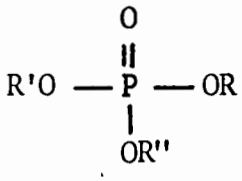
3. PHOSPHORIC AND CARBOXYLIC AMIDES

3.1 Introduction

Biologically, phosphorus is an extremely important element. Generally the phosphorus compounds of biological importance have the phosphoryl structure (1), i.e. tetravalent phosphorus



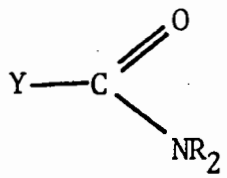
(1)



(2)

with sp^3 hybridization. However, the remaining 2p electrons on the oxygen can now interact with the unfilled d_{xy} and d_{yz} orbitals on the phosphorus resulting in a P = O double bond.[18] Particularly important biologically are the phosphate esters (2).

Although phosphoric amides (4) are found in biological structures,[19] they are not as widespread as the carboxylic amides (3) which are of great importance in biological structures (e.g. in the peptide linkage).[17] This has been explained in terms of the different bonding schemes in these two amide systems. Phosphoric amides are much less stable than



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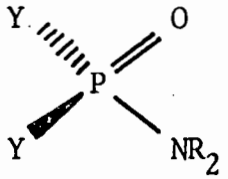
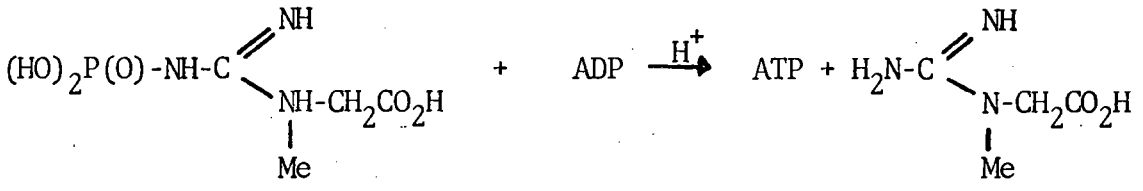


(4)

their carboxylic counterparts particularly under conditions of acid catalysis. In (4) the interaction between the lone pair on the nitrogen atom and the acyl group is much lower than in (3), and accounts for the lower stability of compounds of the type (4).[20,21]

Interest in the nature of P – N bonding in biological structures stems from the importance of the lability of this bond in phosphorylation processes, for example in the reproduction of ATP from ADP in muscle under strain:[22,23]

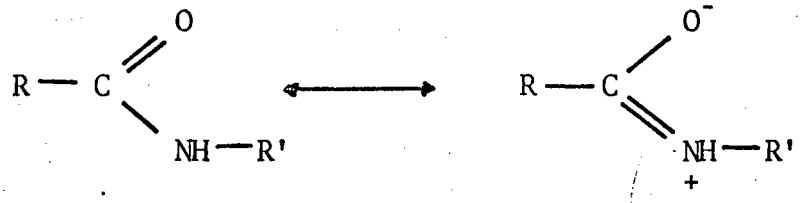


(5) (6)

In addition phosphoramide mustards ($P(O)N(CH_2CH_2Cl)_2$) are under investigation for use in cancer chemotherapy.[24] The phosphoramidates, which are esters of phosphoramidic acid ($(HO)_2P(O)NH_2$), exhibit toxic properties towards mammals and insects and are important as pesticides.[25,26]

3.2 Structural Effects in Carboxylic and Phosphoric Amides

In secondary carboxylic amides (7), the nature of the molecular fragments R and R' determines the detailed structural parameters of the amide linkage. There is a resonance interaction of the lone pair on the

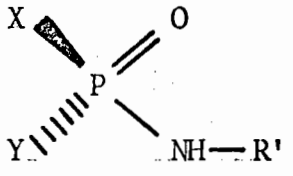


(7) (8)

nitrogen and the acyl group. The nitrogen-acyl rotation barrier in these compounds is greater than 83 kJ $mole^{-1}$,[20] indicating the importance of the contribution of the dipolar resonance structure (8) to the overall structure. The contribution of (8) is however influenced by the nature of the substituents R and R'.

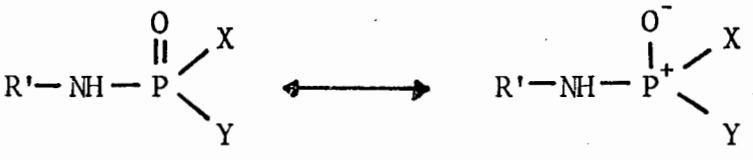
The contribution of (8) to the overall structure and the influence of substituents has been determined from the ν(C=O) I.R. stretching frequencies[27] and n.m.r. spectroscopy.[28] Resonance interactions within the amide group are also influenced by intermolecular hydrogen bonding effects.

Phosphoramidates (9) can be considered as close structural analogues of the carboxylic amides (7). Resonance interactions between the



(9)

phosphoryl group and the lone pair of the nitrogen atom in (9) have been shown to be less marked than in the analogous carbonyl system, and it has been suggested that conjugation is limited to the phosphoryl group (11).[29] Thus the polar affect of the substituent R' would be less



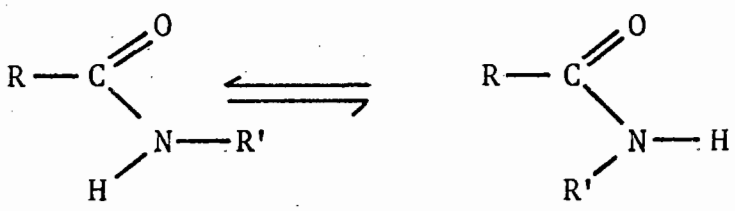
(10) (11)

effectively transmitted to the phosphoryl group in (10) than it would be transmitted to the carbonyl group in (7). In phosphoric amides (4)

the nitrogen-phosphoryl rotational barrier has been determined as not exceeding 33 kJ $mole^{-1}$,[21] which is much lower than the value in the carboxylic amides where extensive conjugation involving the nitrogen atom has been demonstrated. As in the case of the carboxylic amides the resonance effects ((10)↔(11)) would be influenced by intermolecular hydrogen bonding effects.

Hydrogen Bonding

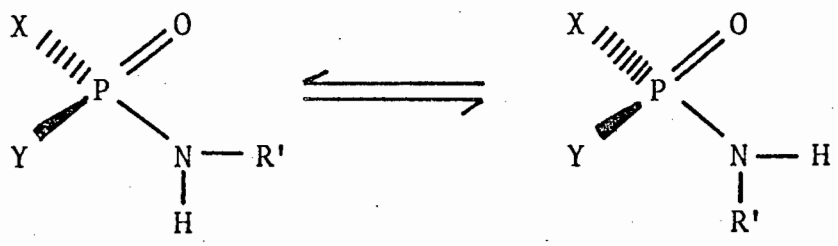
Hydrogen bonding interactions in both carboxylic and phosphoric amides exercise great influence on the resonance interactions in their amidate functions. In the carboxylic amides (7), the hydrogen bond interactions (-N-H....O=C-) are primarily a function of the preferred conformation of the amide group; the *trans* conformation (12) is generally stabilized thermodynamically over the *cis* (13) by a few kJ per mole.[30]



(12) (13)

Intermolecular hydrogen bonding in (12) results in the formation of polymeric aggregates, while (13) is capable of forming hydrogen bonded dimeric pairs.

Since the phosphoryl centre in (9) is not planar but tetrahedral, the *cis* conformation should not suffer the destabilizing effects as the analogous conformation (13). Phosphoric amides should therefore show a greater tendency for the dimerization via intermolecular hydrogen



(14) (15)

bonding than their carboxylic counterparts. This tendency has been demonstrated for these compounds (15) in solution by n.m.r. spectroscopy.[31]

The hydrogen bonding in both carboxylic and phosphoric amides makes the evaluation of substituent effects upon the properties of an amidate function difficult. Spectroscopic studies (e.g. I.R. ν(C=O) stretch or I.R. ν(P=O) stretch) are seldom free of the influence of the -N-H....O= hydrogen bond even in dilute non-polar solutions. This is especially true for the phosphoric amides where hydrogen bonding effects have been demonstrated even in dilute solutions.[32]

Solid State Structure

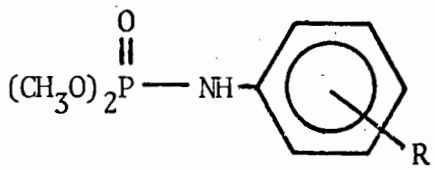
Carboxylic amides have been the subject of a great many X-ray crystallographic studies.[33] This has permitted the detailed analysis of the structural features of the amide function in these compounds. In carboxylic amides the C-N and C=O bond lengths show reciprocal dependence as a consequence of resonance describing the amide function.[34] It is also found that bulky substituents significantly distort the planarity of the amide group thus reducing the resonance interaction in the amide function. The geometry of the amide function is also distorted by

intermolecular hydrogen bonding. In certain peptides it has been found that the amide function suffers significant distortion from planarity in order to achieve an energetically favourable linear intermolecular hydrogen bond.[33]

Structural data on phosphoric amides are sparse and analyses of structural features in these compounds are unavailable. There is much scope for crystallography in this area.

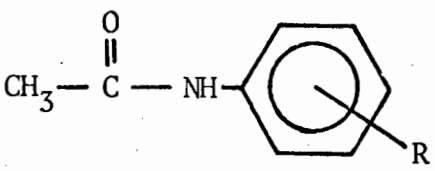
3.3 A Spectrophotometric Study of Phosphoramidates and Acetanilides

Modro and Rijkmans have undertaken a systematic study[35] of a series of ring-substituted phosphoramidates (16) and acetanilides (17).



(16)

R = H; 2-Me; 3-Me; 4-Me; 3,4-di Me; 2,6-di Me; 2-Et; 4-Et; 4 *n*-Bu; 2-*t* Bu; 4-Ph; 3-F; 4-F; 3-OMe; 4-OMe; 3-NO_2, 4-NO_2; 4-NMe_2.



(17)

R = as above (see Table 3.1).

This study involved an investigation of the substituent effects on the vibrational frequencies of the phosphoryl group in relation to the

analogous effects in the carboxylic derivatives (17),[35] and a study of the kinetics of the acid catalysed hydrolysis of these compounds.[36-38]

The results of infrared study are shown in Table 3.1, which gives the ν(P=O) values determined for the series of phosphoramidates (16).[35] In the N-methyl derivative of the parent phosphoramidate (R=H) the ν(PO) band (Nujol) appears at 1275 cm^{-1}, whereas the band in the parent phosphoramidate (R=H) appears at 1229 cm^{-1} (Nujol). Since the N-methyl derivative is not capable of self-association, the difference of 46 cm^{-1} can be taken as a measure of the hydrogen bonding effect upon the stretching frequency of the phosphoryl group.

It is apparent that, for most of the anilides in Table 3.1 hydrogen bonding effects do not disappear when the substrate is transferred from the solid state (Nujol mull) to the dilute solution in benzene or CCl_4. In the substrates substituted only by alkyl groups, electronic effects should be similar; the observed differences are interpreted primarily in terms of the degree of hydrogen bonding. This also appears to be true for all *meta*- and *para*-mono-substituted derivatives (except *p*-*t*-Bu), as indicated by the low values of the corresponding $\nu_{(CCl_4)} - \nu_{(Nujol)}$ shifts. The average value of this shift for compounds (1a, 1c, 1d, 1h, 1i, and 1ℓ - 1s) is -0.6 cm^{-1}, in contrast to the corresponding value of *ca.* +60 cm^{-1} observed for carboxylic anilides.[27]

It is proposed that the substrates characterised by the low value of the $\nu_{(CCl_4)} - \nu_{(Nujol)}$ shifts exist primarily as dimers (18) even at low concentration; this would account for the small effect of solvent on ν(PO) in the phosphoramidates. In carboxylic amides the hydrogen

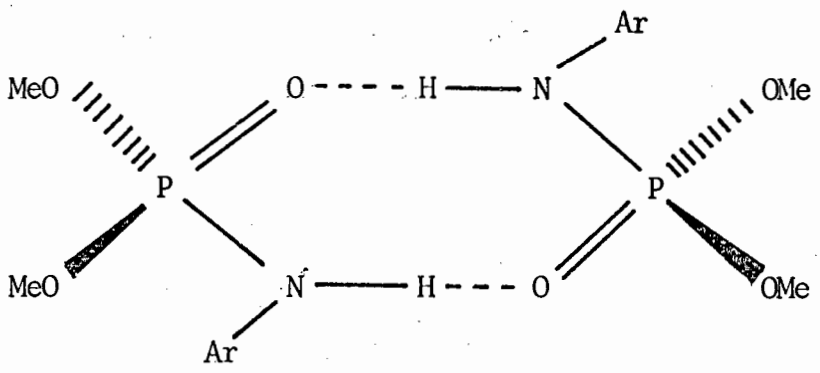
Table 3.1 Values for ν_{PO}(str) (in cm^{-1}) for Phosphoramidates (from Modro *et al.*[35])

	X in (16)	In nujol		In benzene[a]		In CCl_4[a]		
		ν_{PO}	$\Delta\nu_{PO}$[b]	ν_{PO}	$\Delta\nu_{PO}$[b]	ν_{PO}	$\Delta\nu_{PO}$	$\nu_{(CCl_4)}-\nu_{(nujol)}$
1a	H	1229.0	0.0	1228.0	0.0	1228.5	0.0	-0.5
1b	2-Me	1238.5	+9.5	1242.0	+14.0	1249.5	+21.0	+11.0
1c	3-Me	1235.0	+6.0	1235.0	+ 7.0	1238.0	+9.5	+3.0
1d	4-Me	1231.5	+2.5	1228.5	+ 0.5	1228.0	-0.5	-3.5
1e	3,4-diMe	1237.0	+8.0	1235.0	+ 7.0	1225.0	+26.5	+18.0
1f	2,6-diMe	1237.0	+8.0	1272.5	+44.5	1273.0	+44.5	+36.0
1g	2-Et	1242.0	+13.0	1237.0	+9.0	1266.0	+37.5	+24.0
1h	4-Et	1227.5	-1.5	1218.5	-9.5	1218.0	-10.5	-9.5
1i	4*n*-Bu	1232.0	+3.0	1227.0	-1.0	1227.5	-1.0	-4.5
1j	4*t*-Bu	1235.5	+6.5	1229.0	+1.0	1254.0	+25.5	+18.5
1k	2*t*-Bu	c	-	1251.5	+23.5	1275.0	+46.5	-
1ℓ	4-Ph	1225.5	-3.5	1227.0	-1.0	1227.5	-1.0	+2.0
1m	3-F	1233.0	+4.0	1235.5	+7.5	1235.5	+7.0	+2.5
1n	4-F	1228.5	-0.5	1225.5	+2.5	1229.0	+0.5	+0.5
1o	3-OMe	1233.5	+4.5	1236.0	+8.0	1235.5	+7.0	+2.0
1p	4-OMe	1224.0	-5.0	1225.0	-3.0	1225.0	-3.5	+1.0
1q	3-NO_2	1236.0	+7.0	1234.0	+6.0	1233.5	+5.0	-2.5
1v	4-NO_2	1231.5	+2.5	1234.0	+6.0	1234.6	+6.0	+3.0
1s	4-NMe_2	1218.0	-11.0	1219.0	-9.0	1217.0	-11.5	-1.0

a 0.1 % solution

b $\Delta\nu_{PO} = \nu_{PO}(x) - \nu_{PO}(H)$

c No homogenous mull could be prepared.



(18)

bonding results in polymeric aggregation which is solvent dependent. In substrates substituted by *ortho*-alkyl groups large $\nu_{(CCl_4)} - \nu_{(Nujol)}$ shifts are found. The presence of an *ortho*-substituent introduces significant steric crowding which restricts the formation of dimers (18); hence the greater solvent dependence of $\nu(P{=}O)$.

Figure 3.1 shows a plot of $\Delta\nu_{PO}$ (expressed relative to the unsubstituted anilide, $\Delta\nu_{PO}/\nu_{O}$, against Hammet's σ constants for a few representative anilides. In the acetanilides the $\nu(C{=}O)$ stretches are considered to be free of hydrogen bonding effects; this cannot be said of the phosphoramidates. It is however proposed that since the phosphoramidates in Figure 3.1 exist largely as self-associated species hydrogen bonding effects should be similar, since any polar effects of the substituents introduced to the aromatic moiety would change the hydrogen donor property of the phosphoryl oxygen and the acceptor property of the NH group in opposite directions.

It is apparent from Figure 3.1 that the effect of the polarity of the substituent is less effectively transmitted to the P=O bond in the phosphoramidates than it is transmitted to the C=O bond in the acetanilides.

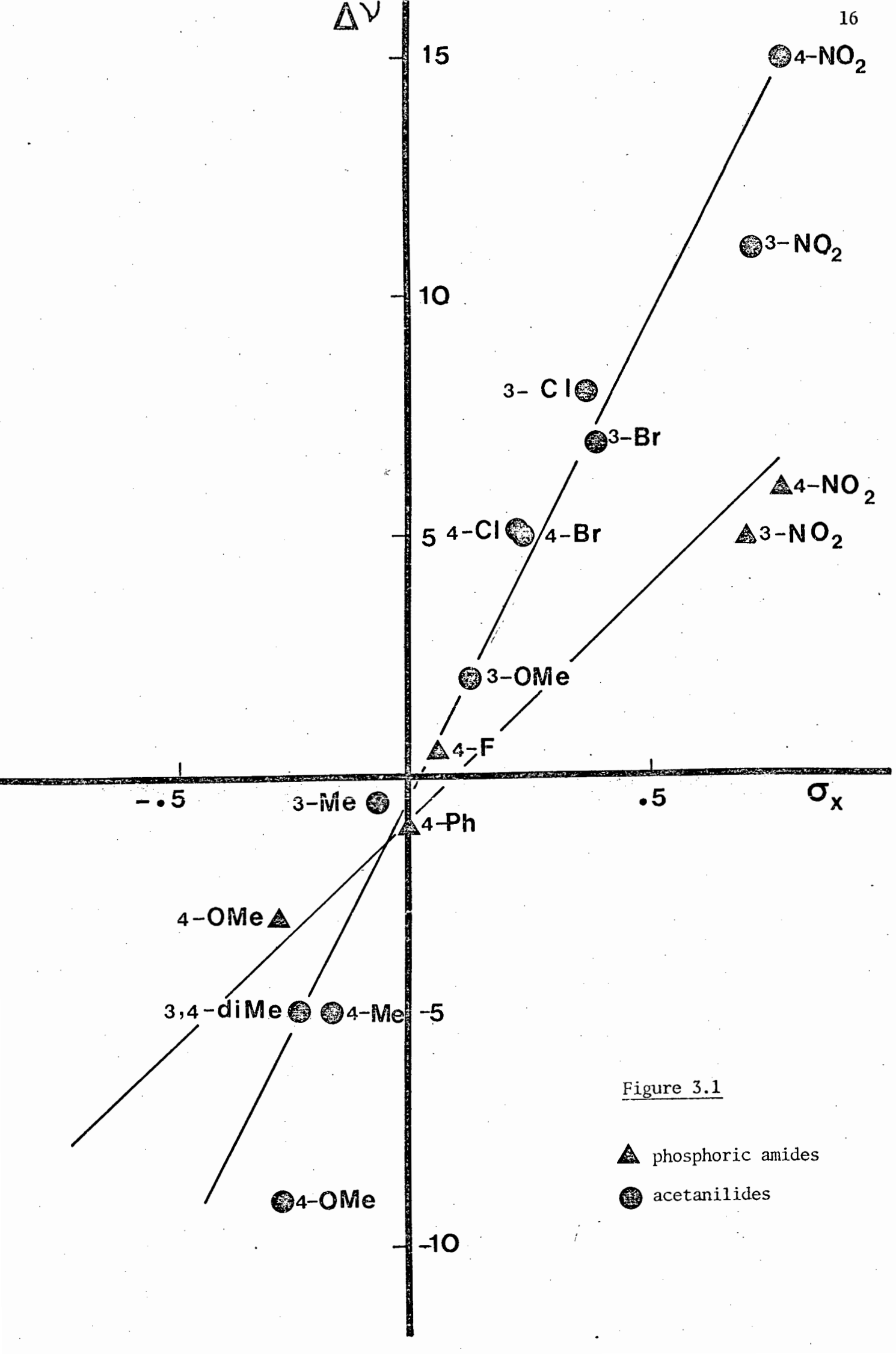
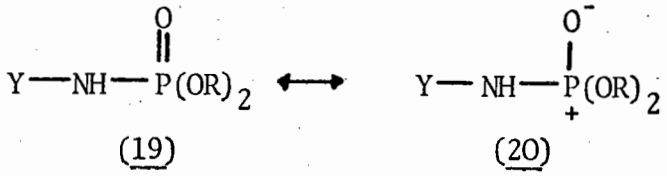

Δν
15
10
5
-5
-10
-.5
.5
σx
4-NO2
3-NO2
3-Cl
3-Br
4-NO2
3-NO2
4-Cl
4-Br
3-OMe
4-F
3-Me
4-Ph
4-OMe
3,4-diMe
4-Me
4-OMe
phosphoric amides
acetanilides

Figure 3.1

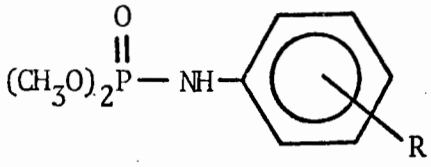
This is evidence that resonance in the phosphoric amides is restricted to the phosphoryl group as proposed earlier (19,20)[28,29].



(19) (20)

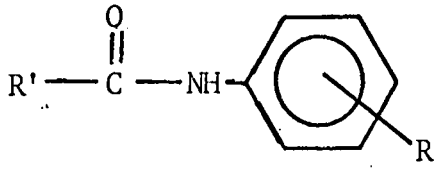
3.4 Objectives

The results above indicate that the factors influencing the amide function in the phosphoramidates are complex. It is however convenient that only small differences are observed between the solid state and the solution properties of some of the phosphoramidates. The determination of the solid state structure of these compounds should reveal useful information about the structure in solution as well. Consequently the crystal structures of a representative number of the phosphoramidates listed in Table 3.1 and their related carboxylic amides were determined.



(16)

1. R = *p*-OCH_3
2. = *p*-NO_2
3. = H
4. = *o*-Et
5. = 2,6-di Me



(17)

6. R = *p*-OCH_3, R' = CH_3
7. = *p*-OCH_3, = Ph
8. = *p*-NO_2, = Ph.

The aims of this part of the research project are:

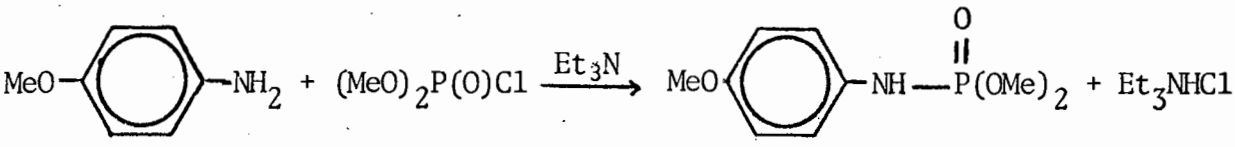
1. to investigate the solid state structures of some phosphoramides and related carboxylic amides.
2. to correlate the solid state data of the phosphoramides with previously determined spectroscopic data.
3. to compare the bonding in phosphoric and carboxylic amides.

CHAPTER FOUR

4. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIMETHYL-N-(*p*-METHOXYPHENYL)-PHOSPHORAMIDATE

4.1 Experimental

The title compound was synthesized[38] according to the reaction:



Freshly distilled dimethylphosphorochloridate (0.035 mole) in 20 ml of dry benzene was added dropwise to a stirred solution of the freshly distilled substituted aniline (0.035 mole) and triethylamine (0.040 mole) in 80 ml dry benzene. Moisture was excluded from the reaction mixture and the temperature was kept below 15°C during addition. The mixture was stirred for a further 3-5 hours at room temperature and allowed to stand overnight. The triethylaminehydrochloride was filtered off and washed with dry benzene, the filtrates combined, washed with distilled water, dried over anhydrous sodium sulphate and the solvent removed under vacuum. The product crystallized from the resulting oil on standing. Suitable single crystals (colourless needles) were obtained from a petroleum ether solution (60-80°C). Found for $C_9H_{14}NO_4P$: C = 46.45 %, H = 6.10 %, N = 5.95 %. Calculated: C = 46.76 %, H = 6.10 %, N = 6.06 %. The density was determined by the flotation in a calibrated density column.[39]

Preliminary oscillation and Weissenberg photography ($0kl$, $h0l$, $hk0$ and hkl) indicated the monoclinic space group $P2_1/n$. Inspection of the diffracto-

meter data revealed systematic absences consistent with the space group $P2_1/n$. This is a non-standard setting of the space group $P2_1/c$.[11] The relationship between the space groups $P2_1/c$ and $P2_1/n$ is shown in Figure 4.1. The structure was refined in the space group $P2_1/n$. The diffractometer data were not corrected for absorption because the absorption correction factor A* remained equal to 1.0 over the entire range of θ values scanned by the diffractometer.

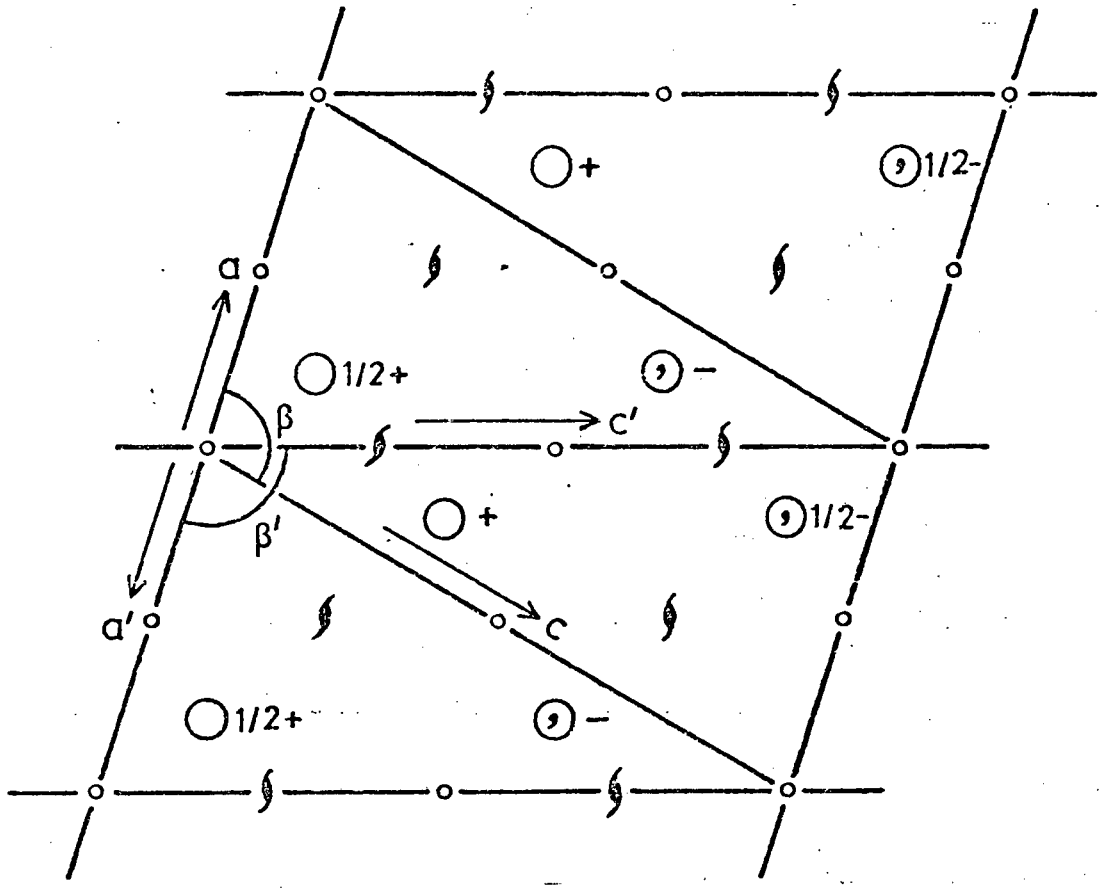


FIGURE 4.1: The relationship (not drawn to scale) between the unit cell (bounded by a, b, and c) corresponding to the space group $P2_1/n$, and the unit cell (bounded by a', b', and c') corresponding to the space group $P2_1/c$. The equivalent positions are:

$P2_1/n$: x,y,z; $-x,-y,-z$; $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$

$P2_1/c$: x,y,z; $-x,-y,-z$; $x, \frac{1}{2}-y, \frac{1}{2}+z$; $-x, \frac{1}{2}+y, \frac{1}{2}-z$.

The two cells are projected onto (010).

Table 4.1 Crystal Data and Experimental and Refinement Parameters for the Structure Analysis.

Crystal Data	
Molecular formula	$C_9H_{14}NO_4P$
M_r	231.2
Space Group	$P2_1/n$
a	21.10(1) Å
b	11.018(6) Å
c	9.871(5) Å
β	95.03(2)°
V	2285.7 Å^3
D_m	1.33 Mg m^{-3}
D_c	1.34 Mgm^{-3} for Z = 8.
μ(Mo-K_α)	0.189 mm^{-1}
μR min., μR max.	0.02, 0.04
A*	1.0
F(000)	976
Data Collection	
Crystal dimensions	0.4 x 0.25 x 0.20 mm
Scan mode	ω-2θ
Scan width	0.96° θ
Scan width	0.03° θs^{-1}
Range scanned (2θ)	6° - 40°
Stability of standard reflections	1.45 %
Number of reflections collected	2279
Number of 'observed' reflections	1567 with $I_{rel} > 2\sigma\ I_{rel}$
Final Refinement	
Number of variables	298
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.048
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.041
Weighting scheme w	$(\sigma^2 F)^{-1}$
U_{iso} (aromatic H)	0.075(7) Å^2
U_{iso} (methyl H)	0.163(8) Å^2
U_{iso} (amide H)	0.09(2) Å^2

4.2 Solution and Refinement of the Structure

The structure was solved by the automatic centrosymmetric direct methods routine of the SHELX program system,[12] in which an *E*-map yielded the positions of most of the heavy atoms. After a number of cycles of least-squares refinement a difference electron-density map was calculated which revealed the positions of the remaining heavy atoms. The positions of most of the hydrogen atoms including the amide hydrogen atom were located in a subsequent difference electron-density map calculated after further cycles of least-squares refinement. The final refinement was carried out with the heavy atoms treated anisotropically, with the methyl hydrogen atoms refined as a rigid group and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atoms were constrained at 1.00 ± 0.01 Å from their respective nitrogen atoms. The isotropic temperature factors of the hydrogen atoms were treated as three single parameters. Details of the final refinements are listed in Table 4.1. In the final cycle of least-squares refinement the average shift to error ratio was less than 1 %. A final difference electron-density map calculated after the final least-squares refinement revealed no peaks > 0.12 e $Å^{-3}$. An analysis of variance calculated after the final refinement is listed in Table 4.2. The final fractional atomic coordinates and temperature factors* are given in Tables 4.3, 4.4 and 4.5. Lists of observed and calculated structure factors are given in Appendix 4.

* All anisotropic temperature factors are of the form

$$T = \exp[-2\pi^2(U11h^2a^{*2} + U22k^2b^{*2} + U33l^2c^{*2} + 2U23klb^*c^* + 2U13hla^*c^* + 2U12hka^*b^*)]$$

Table 4.2 Analysis of Variance[a]

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	243	155	198	190	159	230	193	199	1567
V	131	126	138	113	132	145	125	131	131

(b) As a function of sin (θ)

sin(θ)	0	.15	.20	.23	.25	.27	.29	.30	.32	.34	.38
N	159	204	167	145	160	179	100	200	165	88	
V	212	152	121	109	106	111	114	107	111	118	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.18	.20	.22	.24	.26	.29	.31	.35	.41	1.00
N	183	154	147	154	157	211	102	156	159	144	
V	106	118	111	114	117	133	113	127	141	205	

(d) As a function of |Miller index|

$\lvert h\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	61	120	123	119	117	112	108	103	102	96	92	73	70	65	206
V	133	159	156	125	134	142	138	128	113	127	143	115	99	106	113

$\lvert k\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	130	216	208	214	190	178	154	117	89	55	16	0	0	0	0
V	152	159	161	120	121	110	108	105	106	105	136	0	0	0	0

$\lvert l\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	116	259	245	228	209	173	154	105	62	16	0	0	0	0	0
V	126	150	145	135	120	120	115	118	106	87	0	0	0	0	0

[a] N = Number of reflections in the group; $V = 100[M(\Sigma w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 4.3 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	Molecule A			Molecule B		
	x	y	z	x	y	z
P(1)	1708(1)	2694(2)	-2890(2)	1248(1)	223(1)	559(2)
O(1)	2108(2)	2435(3)	-1638(4)	805(2)	860(3)	-425(4)
O(2)	1845(2)	1891(4)	-4177(4)	1327(2)	-1173(3)	316(4)
O(3)	1795(2)	3986(4)	-3485(4)	1043(2)	215(3)	2063(4)
O(4)	-1049(2)	2258(4)	-6764(4)	4248(2)	-231(4)	3647(5)
N(1)	962(2)	2437(4)	-2695(5)	1964(2)	782(4)	582(5)
C(1)	453(3)	2426(6)	-3734(6)	2524(3)	488(5)	1409(6)
C(2)	379(3)	3268(5)	-4785(7)	3084(3)	1079(5)	1175(6)
C(3)	-125(3)	3186(6)	-5772(6)	3639(3)	824(6)	1931(6)
C(4)	-575(3)	2277(6)	-5710(6)	3662(3)	-40(6)	2955(6)
C(5)	-517(3)	1467(6)	-4655(7)	3104(3)	-631(5)	3213(6)
C(6)	-7(3)	1526(6)	-3679(6)	2541(3)	-374(5)	2435(6)
C(7)	2382(3)	2115(7)	-4946(7)	782(3)	-1977(6)	234(7)
C(8)	1852(4)	5050(6)	-2605(8)	846(4)	1341(6)	2660(6)
C(9)	-1459(3)	1214(6)	-6845(7)	4289(3)	-938(6)	4828(7)

Table 4.4 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms.

	Molecule A			Molecule B		
	x	y	z	x	y	z
H(1)	91	186	-194	200	144	-10
H(21)	72	399	-483	307	175	38
H(31)	-18	384	-659	407	129	174
H(51)	-88	77	-460	311	-129	402
H(61)	4	87	-286	211	-85	263
H(71)	238	136	-564	89	-290	-2
H(72)	284	218	-438	39	-164	-45
H(73)	229	294	-552	65	-194	127
H(81)	195	588	-313	76	107	368
H(82)	205	511	-156	42	171	214
H(83)	134	492	-262	122	203	272
H(91)	-175	130	-780	477	-96	530
H(92)	-176	127	-601	413	-185	460
H(93)	-122	35	-681	398	-53	552

Table 4.5 Anisotropic Temperature Factors ($Å^2 \times 10^3$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
			Molecule A			
P(1)	59(1)	53(1)	53(1)	9(1)	7(1)	-7(1)
O(1)	53(3)	70(3)	54(3)	23(2)	3(2)	-9(2)
O(2)	79(3)	80(3)	70(3)	-11(3)	26(3)	-7(3)
O(3)	84(4)	65(3)	80(3)	25(3)	-2(3)	-18(3)
O(4)	71(3)	79(4)	61(3)	3(3)	-6(3)	14(3)
N(1)	43(3)	63(4)	46(3)	12(3)	11(3)	-4(3)
C(1)	46(4)	48(4)	39(4)	-5(4)	14(3)	3(4)
C(2)	61(5)	44(4)	58(5)	11(4)	12(4)	0(4)
C(3)	64(5)	54(5)	51(5)	8(4)	6(4)	12(4)
C(4)	46(5)	58(5)	48(5)	-7(4)	6(4)	-7(4)
C(5)	53(5)	58(5)	57(4)	10(4)	12(4)	4(4)
C(6)	54(4)	65(5)	42(4)	11(4)	6(4)	-3(4)
C(7)	96(6)	113(6)	85(6)	-2(5)	60(5)	-6(5)
C(8)	149(7)	44(5)	99(6)	-18(5)	9(5)	-5(5)
C(9)	60(5)	92(6)	79(5)	-14(5)	-4(4)	-1(5)
			Molecule B			
P(1)	51(1)	54(1)	46(1)	6(1)	12(1)	3(1)
O(1)	47(3)	69(3)	53(3)	18(2)	13(2)	9(2)
O(2)	60(3)	50(3)	60(3)	-3(2)	17(2)	4(2)
O(3)	63(3)	65(3)	44(3)	7(2)	27(2)	3(2)
O(4)	65(3)	107(4)	73(3)	36(3)	-5(3)	-11(3)
N(1)	46(4)	54(4)	51(4)	14(3)	9(3)	-4(3)
C(1)	51(5)	47(4)	39(4)	-1(3)	8(3)	-1(4)
C(2)	60(5)	61(4)	44(4)	15(4)	2(4)	-13(4)
C(3)	55(5)	76(5)	59(5)	19(4)	0(4)	-23(4)
C(4)	53(5)	64(5)	45(4)	8(4)	0(4)	1(4)
C(5)	58(5)	51(4)	47(4)	10(3)	10(4)	-3(4)
C(6)	49(4)	58(4)	51(4)	8(4)	8(4)	-6(4)
C(7)	79(6)	66(5)	105(6)	-7(4)	18(4)	-30(4)
C(8)	130(7)	62(5)	60(5)	-8(4)	37(4)	18(5)
C(9)	78(5)	86(6)	77(6)	18(4)	-11(4)	4(4)

4.3 Description of the Structure and Discussion

Molecular Structure

The principal bond lengths and angles are given in Tables 4.6 and 4.7 respectively and selected torsion angles in Table 4.8. A perspective view of the title compound is given in Figure 4.2, showing the atomic nomenclature.

The compound crystallizes with eight molecules in the unit cell, the asymmetric unit comprising a hydrogen-bonded dimer, associated by a pair of N – H ---- O = P hydrogen bonds which are crystallographically independent. The two independent molecules are labelled A and B respectively. Both molecules crystallize in the *syn*-coplanar orientation of the phosphoryl and N – H groups required for dimerization. The two independent molecules, A and B, have similar conformations although there are important differences between them. The conformation of the groups attached to the phosphorus atom is similar in both molecules (refer to the torsion angles in Table 4.8). However the orientation of the substituted phenyl ring with respect to the phosphoramidate group differs in the two molecules. In molecule A the *p*-methoxy substituent is bent towards the same side of the molecule as the N – H and P = O groups, in molecule B it is orientated in the opposite direction.

The coordination of the phosphorus atom is distorted tetrahedral. Angles of the type O(1) = P – O or N are all greater than the tetrahedral angle of $\approx 109^{\circ}$. This was also observed in O-methyl-O-(2-chloro-4-tertbutyl-phenyl)-N-methyl phosphoramidate (crufomate)[40] and 5,5-dimethyl-2-oxo-2-aminobenzo-1,3,2-dioxaphosphorinane (phosphorinane).[41] The P = O bond

Table 4.6 Principal Bond Lengths (Å) with e.s.d.'s in Parentheses.

Bond	Molecule A	Molecule B
P(1)-O(1)	1.462(2)	1.466(2)
P(1)-O(2)	1.595(2)	1.567(2)
P(1)-O(3)	1.557(2)	1.582(2)
P(1)-N(1)	1.626(2)	1.630(3)
N(1)-C(1)	1.419(4)	1.413(4)
C(1)-C(2)	1.391(4)	1.387(4)
C(2)-C(3)	1.381(4)	1.361(4)
C(3)-C(4)	1.385(4)	1.386(4)
C(4)-C(5)	1.369(4)	1.388(4)
C(5)-C(6)	1.380(4)	1.386(4)
C(6)-C(1)	1.392(4)	1.386(4)
C(4)-O(4)	1.378(4)	1.375(4)
O(2)-C(7)	1.438(4)	1.448(4)
O(3)-C(8)	1.457(4)	1.450(4)
O(4)-C(9)	1.436(4)	1.399(4)

Table 4.7 Principal Bond Angles (°) with e.s.d.'s in Parentheses.

Angle	Molecule A	Molecule B
O(1)-P(1)-O(2)	114.8(1)	116.0(1)
O(1)-P(1)-O(3)	115.7(1)	114.4(1)
O(1)-P(1)-N(1)	111.0(1)	111.3(1)
N(1)-P(1)-O(3)	104.0(1)	108.6(1)
O(2)-P(1)-O(3)	99.8(1)	100.3(1)
N(1)-P(1)-O(2)	110.5(1)	105.2(1)
P(1)-N(1)-C(1)	126.6(2)	130.4(2)
C(6)-C(1)-C(2)	118.1(3)	118.3(3)
C(1)-C(2)-C(3)	120.8(3)	121.1(3)
C(2)-C(3)-C(4)	120.3(3)	121.0(3)
C(3)-C(4)-C(5)	119.3(3)	118.7(3)
C(4)-C(5)-C(6)	120.9(3)	120.1(3)
C(5)-C(6)-C(1)	120.6(3)	120.8(3)
P(1)-O(2)-C(7)	122.1(4)	120.9(2)
P(1)-O(3)-C(8)	121.1(2)	119.2(2)
C(4)-O(4)-C(9)	116.3(3)	119.1(3)

NOTE:

The torsion angle τ(I-J-K-L) is defined as the angle between the vector K-L when viewed down J-K. The sign of τ is positive if J-I is to be rotated clockwise into K-L and negative if counter-clockwise[185].

Table 4.8 Selected Torsion Angles (°) with e.s.d.'s in Parentheses.

Torsion angle	Molecule A	Molecule B
O(1)-P(1)-O(2)-C(7)	-76.9(3)	-56.4(3)
O(3)-P(1)-O(2)-C(7)	46.9(3)	67.4(3)
N(1)-P(1)-O(2)-C(7)	161.1(3)	180.0(3)
O(1)-P(1)-O(3)-C(8)	-42.4(3)	-48.1(3)
O(2)-P(1)-O(3)-C(8)	-166.7(3)	-173.0(3)
N(1)-P(1)-O(3)-C(8)	84.1(3)	77.0(3)
O(1)-P(1)-N(1)-C(1)	-170.6(3)	176.6(3)
O(2)-P(1)-N(1)-C(1)	-45.6(3)	-56.9(3)
O(3)-P(1)-N(1)-C(1)	60.8(3)	49.8(3)
C(9)-O(4)-C(4)-C(3)	170.1(3)	169.7(3)
C(9)-O(4)-C(4)-C(5)	-8.7(3)	-9.7(3)
P(1)-N(1)-C(1)-C(2)	-42.2(3)	177.4(3)
P(1)-N(1)-C(1)-C(6)	142.4(3)	-1.9(3)
N(1)-C(1)-C(2)-C(3)	179.0(3)	180.0(3)
C(6)-C(1)-C(2)-C(3)	-5.5(3)	0.0(3)
N(1)-C(1)-C(6)-C(5)	-176.4(3)	180.0(3)
C(2)-C(1)-C(6)-C(5)	7.9(3)	0.0(3)
C(1)-C(2)-C(3)-C(4)	1.6(3)	0.0(3)
C(2)-C(3)-C(4)-O(4)	-178.4(3)	180.0(3)
C(2)-C(3)-C(4)-C(5)	0.0(3)	-1.0(3)
O(4)-C(4)-C(5)-C(6)	179.1(3)	-179.2(3)
C(3)-C(4)-C(5)-C(6)	2.4(3)	1.4(3)
C(4)-C(5)-C(6)-C(1)	-6.4(3)	-1.2(3)

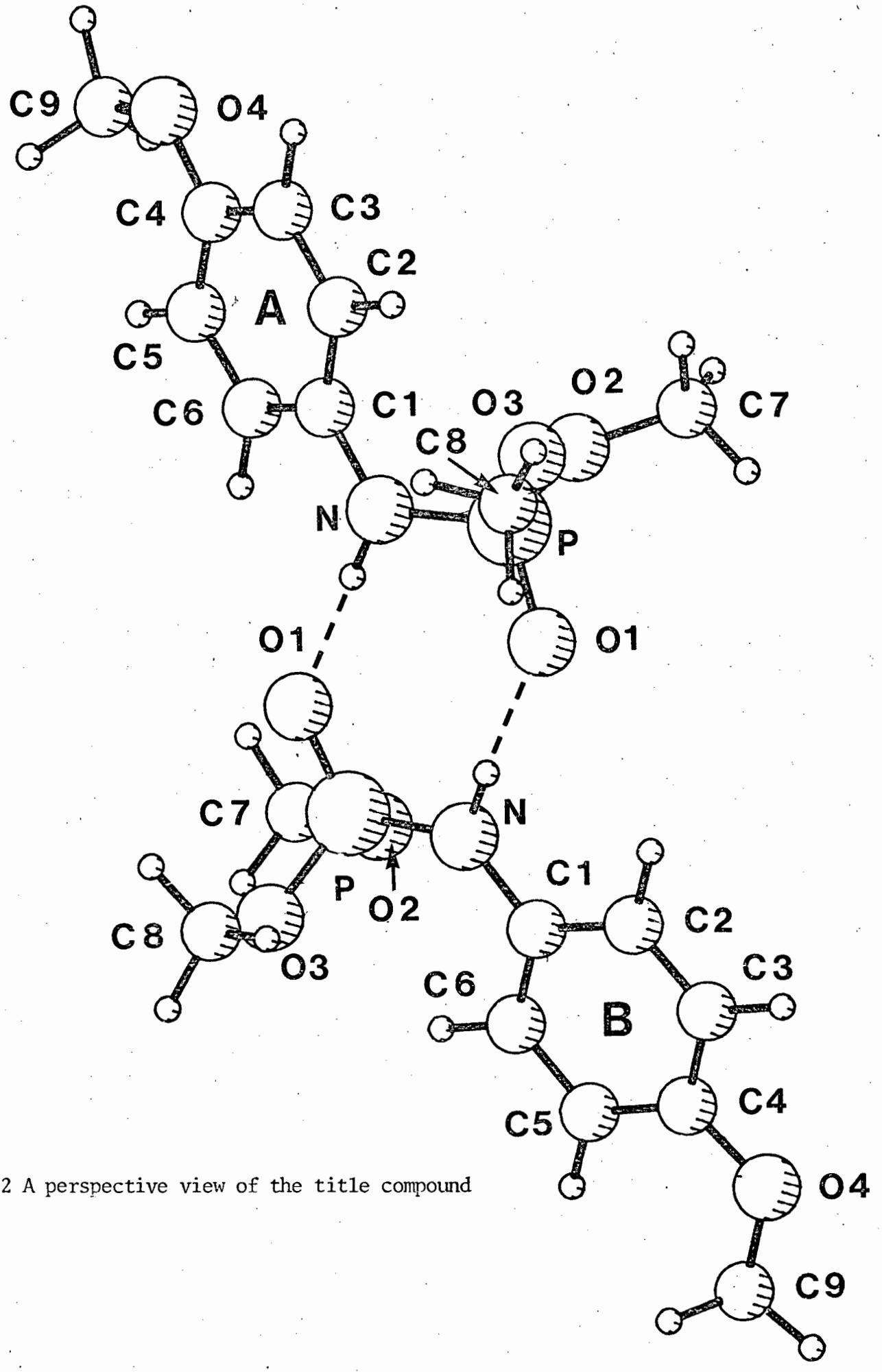


Figure 4.2 A perspective view of the title compound

length is shorter than the value of 1.476(10) Å found in the N-phenyl derivative (phosphorinane)[41] and similar to the value of 1.459(4) Å found in crufomate.[40] The P – O bond lengths are within the ranges normally observed.[42] The P – N bond distances are shorter than the value of ≈ 1.77 Å associated with a P – N single bond,[43] but longer than the upper limit of ≈ 1.60 Å found in cyclotriphosphazenes[44] where there is thought to be a strong pπ - dπ delocalization along the P – N bond. The N – C(1) bond length is shorter than the value of 1.413(3) Å found in acetanilide,[45] indicating possible interaction of the nitrogen atom with the phenyl ring.

The phosphoramidate group is approximately planar as indicated by the O(1) – P – N – C(1) torsion angles of -170.6(3)° and 176.6(3)° for molecules A and B respectively. The bond angles involving nitrogen (P – N – C(1)) are 126.6(2)° and 130.4(2)°; somewhat larger than the value of 120° expected for sp^2 hybridized nitrogen. The C(2) – C(1) – N(1) – P torsion angles are -42.2(3)° and 177.4(3)° indicating the degree to which the phenyl ring is twisted out of the plane of the phosphoramidate group. The large differences in the magnitude of the torsion angles arise from the arbitrary way the two benzene rings are labelled. In molecule B the twist of the phosphoramidate group with respect to the benzene ring is more apparent from the C(6) – C(1) – N(1) – P(1) torsion angle of -1.9(3)°. This would reduce the π - interaction of the phenyl ring with the nitrogen atom. The phenyl rings are staggered with respect to the CH_3O – P – OCH_3 groups, minimising steric interactions between the N-aromatic groups and the two phosphate ester groups.

The Benzene Ring Geometry

Based on an analysis of the structures of many substituted benzene derivatives it has been found that substitution induces significant distortion of the geometry of the benzene ring.[46-48] In particular the *ipso* angle, α, is sensitive to the polarity of the substituent. These distortions are an indication of the interaction of the substituent with the benzene ring.

In the title compound the α_{OCH_3} angles (C(3) – C(4) – C(5)), 119.3(3)° and 118.7(3)°, are similar to the mean value of 119.9(1)° derived from many structural results on *para*-substituted anisoles.[48] In *p*-methoxybenzoic acid α_{OCH_3} = 120.2(2)°,[49] somewhat longer than in the title compound. The α_{NH} angles (C(6) -- C(1) -- C(2)) are 118.1(3)° and 118.3(3)°, slightly shorter than the mean value of 119.1(2)°, based on many derivatives of aniline (Ph -- NH – X).[50] For a substituent capable of sharing π-electrons with the ring, α increases with increasing conjugation.[48] The values of α_{OCH_3} and α_{NH} above therefore indicate that there is significant π interaction of both the methoxy groups and the nitrogen atoms with their respective benzene rings.

These interactions are also mirrored in the deviations of the benzene rings from planarity. Table 4.9 gives the deviations from planarity of the benzene ring in terms of the least-squares plane calculated through the atoms C(1), C(2), C(3), C(4), C(5) and C(6). These deviations are small and a number of the deviations are significant in terms of the estimated standard deviations calculated by the program SHELX. In both molecules the methoxy oxygen atom, O(4), and the amide nitrogen atom are bent out of the least-squares plane calculated through the six-membered

ring. In both molecules the mean length of the central C – C bonds of the rings (C(2) – C(3) and C(5) – C(6)) are 1.380(5) Å and 1.373(5) Å. This is shorter than the mean length of the other four bonds 1.384(6) and 1.387(6) Å (for molecules A and B respectively). This quinonoidal shortening of the central C – C bonds of the ring has been observed in benzene derivatives having a π – donor *para* to a π-acceptor.[49]

The geometry of the *p*-methoxy groups is normal. The oxygen atoms are bent out of the planes of the rings. The C(9) – O(4) bond lengths of 1.436(4) Å and 1.399(4) Å are similar to that found in *p*-methoxybenzoic acid (1.435(3) Å).[49] The C(4) – O(4) bond lengths, 1.378(4) Å and 1.375(4) Å, are longer than the values found in *p*-methoxybenzoic acid[49] (1.356(4) Å), *p*-ethoxybenzoic acid (1.358(3) Å)[51] and 4-isonitroso-acetanilide (1.367(2) Å).[52,53]

Hydrogen Bonding

The hydrogen bonding in the title compound is of particular interest and is the dominant feature of the structure. The compound crystallises as a dimer, associated by a pair of crystallographically independent N – H ---- O = P hydrogen bonds. The dimeric structure was suspected from both the solid state as well as the solution infrared spectrum of this compound (refer to the previous chapter). The details of the hydrogen bonding scheme are given in Table 4.10.

The N ---- O = P values in this compound indicate a hydrogen bond of moderate strength. The values of 2.877(4) Å and 2.886(4) Å can be classed

Table 4.9 The Least-Squares Planes Calculated through the Benzene Rings and the Deviations of Selected Atoms from the Planes with e.s.d.'s in Parentheses.

Equations of the planes:

Molecule A: $-12.334x + 6.575y + 5.9102z = -1.160$

Molecule B: $-5.448x + 7.836y + 6.653z = -0.054$

Deviations (Å):

	Molecule A	Molecule B
N	-0.017(4)	-0.016(4)
*C(1)	-0.010(4)	-0.001(4)
*C(2)	0.014(4)	0.001(4)
*C(3)	-0.004(4)	0.002(4)
*C(4)	-0.008(4)	-0.006(4)
*C(5)	0.011(4)	0.007(4)
*C(6)	-0.002(4)	-0.003(4)
O(4)	-0.059(4)	-0.015(4)

*Atoms used in the calculation of the least-squares plane

Table 4.10 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen bonding	
N(1A)-H(1A) [Å]	1.00(1)
N(1B)-H(1B) [Å]	1.00(1)
H(1A).....O(1B) [Å]	1.88(1)
H(1B).....O(1A) [Å]	1.90(1)
N(1A).....O(1B) [Å]	2.877(4)
N(1B).....O(1A) [Å]	2.886(4)
N(1A)-H(1A)-O(1B) [°]	176.1(9)
N(1B)-H(1B)-O(1A) [°]	168.8(9)
Close contacts	
O(4A).....C(8A) [1]	3.446(4)
O(2B).....C(9A) [2]	3.462(4)
C(3A).....C(8B) [3]	3.359(4)
C(9A).....O(1A) [4]	3.393(4)
C(9B).....N(1A) [5]	3.429(4)
O(2B).....C(7A) [6]	3.360(4)

Symmetry code:

[1] -x 1-y -1-z [4] 0.5+x 1.5-y 0.5+z

[2] -x -y -1-z [5] 0.5-x -0.5-y 0.5-z

[3] x y 1+z [6] 0.5-x -0.5-y -0.5+z

as 'medium' in terms of the average range found in N — H ---- O hydrogen bonds (2.6 - 3.3 Å).[54] The conformation of the phosphoramidate groups in the dimer permit the formation of N — H ---- O angles fairly close to 180°; the formation of linear hydrogen bonds is favoured energetically.[55] The hydrogen bonds in the title compound are longer than the length of 2.802(10) Å found in 'phosphorinane'[41] and are similar to the average length of 2.892(3) Å found in diammonium hydrogen phosphate.[56] The former compound represents a *trans* orientation of the NH and P = O groups of the phosphoramidate function. The extent to which the hydrogen bonding influences the bonding in the phosphoramidate function can only be determined if more structural data on related compounds are available; this will be discussed in chapter 11 in relation to the structures of the phosphoramidates discussed in following chapters.

Molecular Packing

A projection of the crystal structure is shown in Figure 4.3. The predominant intermolecular interaction is the hydrogen bonding within the dimer. The packing unit is the dimer and there are no other important molecular interactions. A number of close contacts less than 3.5 Å are listed in Table 4.10. These are unlikely to have an important influence on the structure.

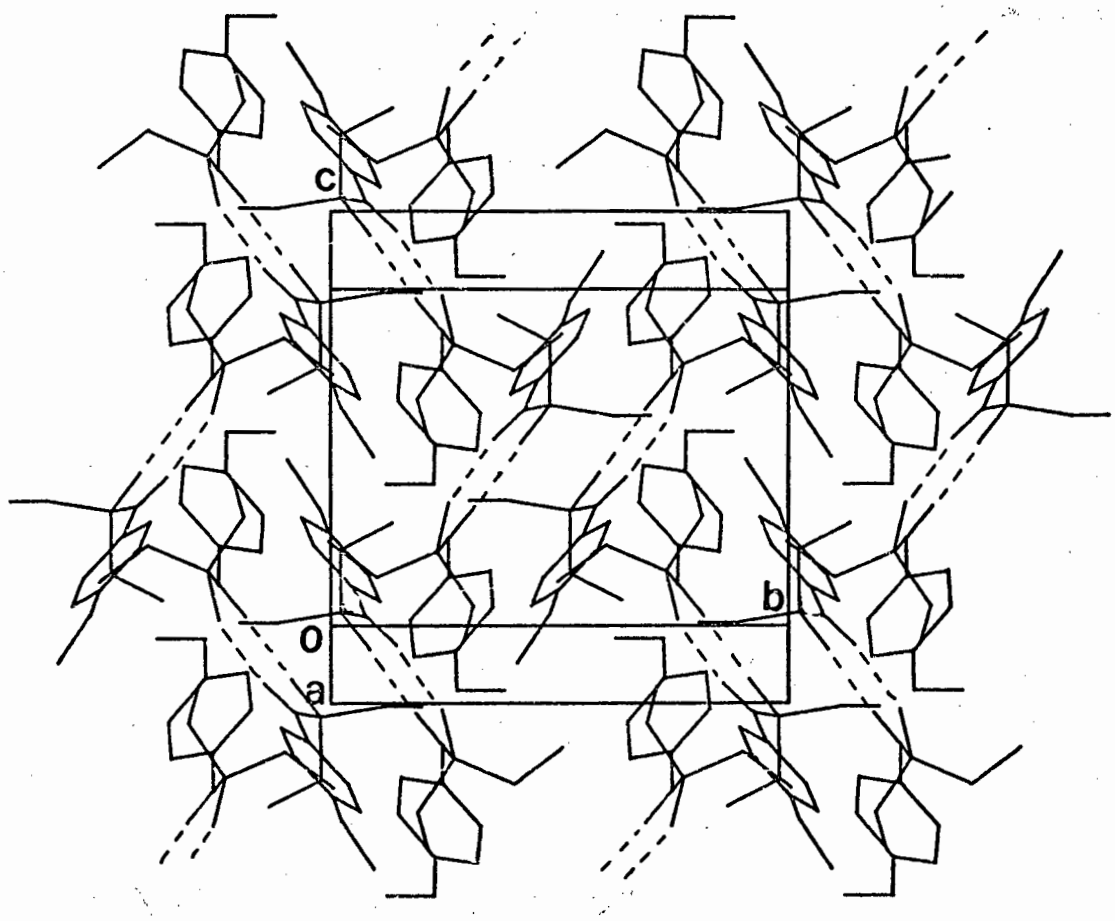


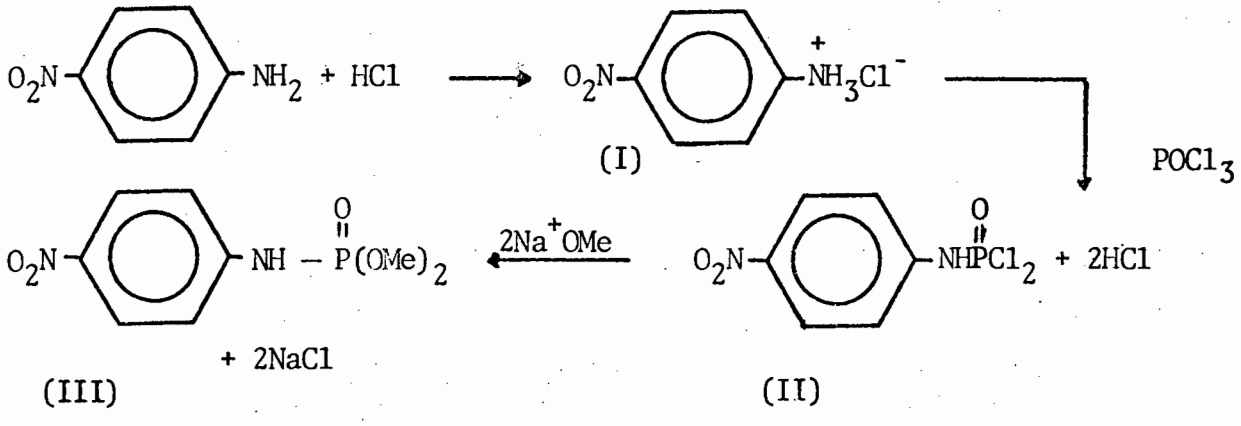
Figure 4.3 A View of the Crystal Packing

CHAPTER FIVE

5. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIMETHYL-N-(*p*-NITROPHENYL)-PHOSPHORAMIDATE

5.1 Experimental

The title compound was synthesized according to the reaction:[38]



The *p*-nitroaniline hydrochloride (I) was obtained as follows. Dry HCl gas was passed through a solution of freshly distilled *p*-nitroaniline (0.065 mole) in 200 ml of dry diethylether. The resulting salt precipitate was filtered, washed with portions of dry diethylether and dried in vacuo over silica gel. The product was not purified further.

The *p*-nitroaniline hydrochloride (0.02 mole) was dissolved in an excess of freshly distilled $POCl_3$ and the mixture stirred under reflux for 5 hours under anhydrous conditions. Thereafter the solution was allowed to stand overnight at room temperature. The excess $POCl_3$ was removed under vacuum and the resulting crude N-(*p*-nitrophenyl) phosphorus oxychloride (II) was obtained as a pale yellow oil.

The crude product (II), approximately 0.025 mole, was dissolved in 25 ml of dry diethylether and added dropwise to a stirred solution of 0.10 mole of Na metal in 200 ml of absolute ethanol, maintained below 20°C. The pH of the solution remained > 7 during addition. The resulting mixture was stirred overnight at room temperature, thereafter the precipitate was filtered and the solvent removed under reduced pressure. The crude product (III) was dissolved in benzene and this solution washed with water, dried over anhydrous sodium sulphate and evaporated to dryness under reduced pressure. The product (III) was recrystallized from a benzene / petroleum ether (60 - 90°C) (4:1) mixture to which a small quantity of chloroform was added. The product crystallized as colourless needles (along *a*). Found for $C_8H_{11}N_2O_5P$: C = 39.05 %, H = 4.45 %, N = 11.45 %; calculated: C = 39.03 %, H = 4.50 %, N = 11.38 %. The density of the compound was determined by flotation in a calibrated density column.[39]

Preliminary oscillation and Weissenberg photography (*h*0*l*, *h*1*l*, *hk*0, *hk*1) indicated the orthorhombic space group *Pbcn*.[11] This choice was subsequently confirmed by inspection of the diffractometer intensity data. The proximity of reflections, due to the relatively large *a* parameter of 24.74 Å, precluded the measurement of background radiation for individual reflections. A background vs. θ curve (obtained from unobserved reflections) was thus used to estimate the background for all the reflections. The third reference reflection used to check crystal stability showed a high percentage standard deviation, indicative of some crystal decay. No absorption correction factor was applied to the diffractometer data as A^* was equal to 1.0 over the scanned range. The crystal details and the details of the diffractometer data collection are given in Table. 5.1.

TABLE 5.1 Crystal Data and Experimental and Refinement Parameters for the Structure Analysis

Crystal Data	
Molecular formula	$C_8H_{11}N_2O_5P$
M_r	246.2
Space Group	*Pbcn*
a	24.71(1) Å
b	7.421(4) Å
c	12.327(6) Å
V	2263.4 Å^2
D_m	1.42 Mgm^{-3}
D_c	1.44 Mgm^{-3} for Z = 8
μ(Mo-$K\alpha$)	0.202 mm^{-1}
μR_{min} , μR_{max}	0.01, 0.04
A*	1.0
F(000)	1024
Date Collection	
Crystal dimensions	0.4 x 0.1 x 0.1 mm
Scan mode	ω - 2θ
Scan width	0.4°θ
Scan speed	0.005°θs^{-1}
Range scanned (2θ)	6-40°
Stability of standard reflections	2.63 %
Number of reflections collected	1263
Number of 'observed' reflections	719 with $I_{rel} > 2\sigma I_{rel}$
Final Refinement	
Number of variables	157
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.075
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.059
Weighting scheme w	$(\sigma^2 F)^{-1}$
U_{iso}(aromatic H)	0.12(2) Å^2
U_{iso}(methyl H)	0.28(3) Å^2
U_{iso}(amide H)	0.16(4) Å^2

5.2 Solution and Refinement of the Structure

The structure was solved by the automatic centrosymmetric direct methods routine of the SHELX[12] program system, in which the first *E*-map yielded the positions of all the heavy atoms. The positions of most of the hydrogen atoms, including the amide hydrogen atom, were located in a difference electron-density map calculated after a number of cycles of least-squares refinement of the atomic positions located in the *E*-map. The final least-squares refinements were carried out with the heavy atoms treated anisotropically, with the methyl hydrogen atoms refined as rigid groups and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atom was constrained at 1.00 ± 0.01 Å from the nitrogen atom. The isotropic temperature factors of the methyl, aromatic and amide hydrogen atoms were treated as three single parameters. In the final cycle of least-squares refinement the average shift to error ratio was less than 1.5 %. A final difference electron density map calculated after the final refinement revealed no peaks of height > 0.2 e $Å^{-3}$. As a final test of the proposed model an analysis of variance was calculated. This is shown in Table 5.2 and shows that the refinement had converged successfully.

Details of the diffractometer data collection and the final refinement is given in Table 5.1. Lists of observed and calculated structure factors are given in Appendix 5. Final atomic parameters and temperature factors are found in Tables 5.3, 5.4 and 5.5.

Table 5.2 Analysis of Variance[a]

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	139	99	75	97	75	59	87	88	719
V	207	187	168	156	151	168	202	182	182

(b) As a function of sin (θ)

sin(θ)	0	.15	.19	.22	.24	.26	.27	.29	.31	.32	.35
N	77	73	79	65	91	46	90	111	34	53	
V	275	160	168	153	161	194	149	189	165	158	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.19	.20	.22	.24	.25	.27	.30	.35	.43	1.00
N	112	42	93	84	37	74	65	68	75	69	
V	189	178	142	149	165	165	149	178	168	292	

(d) As a function of |Miller index|

$\lvert h\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	29	42	50	42	51	43	49	45	40	40	40	34	35	28	151
V	261	260	232	186	179	168	167	169	168	162	161	139	160	128	164

$\lvert k\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	96	162	141	104	94	80	41	1	0	0	0	0	0	0	0
V	237	191	154	144	165	190	193	7	0	0	0	0	0	0	0

$\lvert l\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	59	89	108	77	94	67	78	48	51	26	20	2	0	0	0
V	175	185	205	195	194	171	149	159	176	142	193	291	0	0	0

[a] N = Number of reflections in the group; $V = 100[M(\Sigma w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 5.3 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	x	y	z
P(1)	3099(1)	1889(4)	2043(2)
O(1)	2533(2)	2069(7)	2366(5)
O(2)	3533(2)	2857(8)	2756(6)
O(3)	3263(3)	2827(10)	960(7)
O(4)	5617(3)	-2687(15)	727(7)
O(5)	5246(3)	-5270(13)	1141(6)
N(1)	3258(2)	-257(9)	1972(7)
N(2)	5230(5)	-3596(16)	1008(8)
C(1)	3758(4)	-1050(15)	1719(7)
C(2)	4209(4)	-21(15)	1468(7)
C(3)	4700(5)	-869(19)	1229(7)
C(4)	4710(4)	-2711(21)	1251(7)
C(5)	4273(5)	-3788(15)	1506(7)
C(6)	3791(4)	-2918(16)	1730(7)
C(7)	3586(5)	2404(22)	3888(9)
C(8)	3016(5)	2530(22)	-26(10)

Table 5.4 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen atoms.

	x	y	z
H(1)	294	-107	203
H(21)	419	143	145
H(31)	506	-11	103
H(51)	430	-524	153
H(61)	343	-369	190
H(71)	389	341	407
H(72)	371	112	422
H(73)	321	279	425
H(81)	316	350	-61
H(82)	273	317	51
H(83)	282	144	-45

Table 5.5 Anisotropic Temperature Factors ($Å^2 \times 10^3$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
P(1)	62(2)	71(2)	98(2)	12(2)	-2(2)	7(2)
O(1)	49(3)	80(5)	145(5)	6(5)	3(4)	11(4)
O(2)	69(4)	80(5)	133(6)	-10(5)	-7(4)	-31(4)
O(3)	96(5)	102(6)	116(6)	30(6)	5(5)	5(5)
O(4)	79(5)	223(12)	104(6)	30(7)	24(5)	20(7)
O(5)	101(6)	156(8)	86(6)	-9(7)	-12(5)	57(6)
N(1)	46(5)	51(5)	106(6)	4(5)	8(5)	6(4)
N(2)	119(10)	125(11)	50(7)	7(7)	-9(7)	43(9)
C(1)	50(7)	64(8)	56(7)	0(6)	0(6)	5(6)
C(2)	42(5)	88(7)	91(8)	2(7)	6(6)	-7(8)
C(3)	87(10)	87(10)	63(8)	13(8)	-2(7)	-3(8)
C(4)	39(7)	120(11)	47(6)	0(8)	6(5)	30(8)
C(5)	71(7)	102(9)	58(7)	-7(6)	-5(6)	13(9)
C(6)	56(7)	71(9)	70(7)	1(7)	2(6)	6(6)
C(7)	136(10)	213(17)	76(9)	20(11)	-1(8)	-46(11)
C(8)	146(11)	216(16)	77(8)	-38(10)	-39(8)	-26(12)

5.3 Description of the Structure and Discussion

A perspective view of the title compound showing the atomic nomenclature is given in Figure 5.1. The principal bond lengths, angles and selected torsion angles are given in Tables 5.6, 5.7 and 5.8 respectively.

Molecular Structure

The title compound crystallizes as a monomer which participates in a polymeric network of N – H ---- O = P intermolecular hydrogen bonds. Although the expected dimerization does not occur, the phosphoric amide group still exhibits the *syn*-coplanar orientation of the P = O and N – H groups.

The coordination of the phosphorus atom may be described as distorted tetrahedral. The bond angles involving the phosphorus atom deviate from the ∿109° expected for a sp^3 hybridized phosphorus atom. The bond angles involving phosphorus vary between 95.6° and 117.6°. Angles of the type O(1) = P – OCH_3 are greater than 109°; angles of the type O – P – N are about 109°. The O(2) – P – O(3) angle is compressed to 95.6(4)°. Similar effects were observed in the *p*-methoxy analogue, cruformate[40] and phosphorinane[41]. The P = O bond length is not significantly (in terms of the estimated standard deviations calculated by SHELX) different from the average value of 1.464(2) Å found in the *p*-methoxy analogue. The P – N bond length is larger than the average value of 1.628(2) Å found in the *p*-methoxy analogue. The value of 1.643(7) Å in the title compound is intermediate between the value of ∿ 1.77 Å and 1.60 Å associated with a single and double P – N bond respectively.[43,44] On the other hand the N – C(1) bond length is slightly shorter

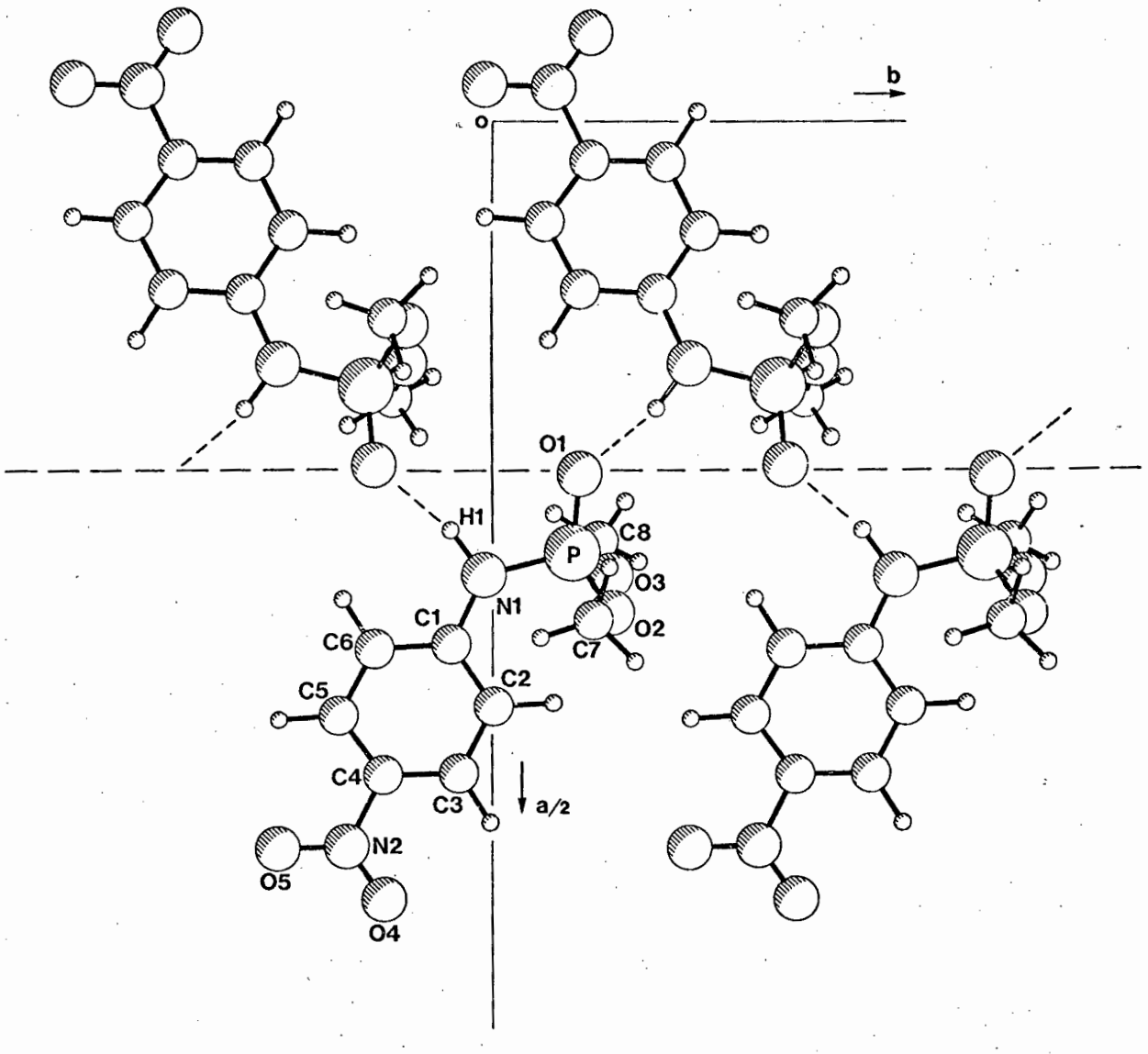


Figure 5.1 A perspective view of the title compound

Table 5.6 Principal Bond Lengths (Å) with e.s.d.'s in Parentheses.

P(1)-O(1)	1.462(5)
P(1)-O(2)	1.563(6)
P(1)-O(3)	1.559(7)
P(1)-N(1)	1.643(7)
N(1)-C(1)	1.406(9)
C(1)-C(2)	1.387(11)
C(2)-C(3)	1.400(11)
C(3)-C(4)	1.367(12)
C(4)-C(5)	1.380(12)
C(5)-C(6)	1.386(11)
C(6)-C(1)	1.388(11)
C(7)-O(2)	1.442(10)
C(8)-O(3)	1.378(11)
N(2)-C(4)	1.475(12)
N(2)-O(4)	1.223(11)
N(2)-O(5)	1.254(11)

Table 5.7 Principal Bond Angles (°) with e.s.d.'s in Parentheses.

O(1)-P(1)-O(2)	117.6(4)
O(1)-P(1)-O(3)	116.2(3)
O(2)-P(1)-O(3)	95.6(4)
N(1)-P(1)-O(1)	109.4(4)
N(1)-P(1)-O(2)	108.1(4)
N(1)-P(1)-O(3)	109.0(4)
P(1)-N(1)-C(1)	128.9(6)
N(1)-C(1)-C(2)	121.8(10)
N(1)-C(1)-C(6)	117.9(10)
C(6)-C(1)-C(2)	120.3(9)
C(1)-C(2)-C(3)	119.9(10)
C(2)-C(3)-C(4)	117.4(10)
C(3)-C(4)-C(5)	124.8(10)
C(4)-C(5)-C(6)	116.7(10)
C(5)-C(6)-C(1)	120.9(9)
N(2)-C(4)-C(3)	117.1(13)
N(2)-C(4)-C(5)	118.1(12)
O(4)-N(2)-O(5)	124.0(12)
C(4)-N(2)-O(4)	119.7(11)
C(4)-N(2)-O(5)	116.2(12)
P(1)-O(2)-C(7)	119.9(7)
P(1)-O(3)-C(8)	124.6(8)

Table 5.8 Selected Torsion Angles (°) with e.s.d.'s in Parentheses.

O(1)-P(1)-O(2)-C(7)	59.8(9)
O(3)-P(1)-O(2)-C(7)	-176.8(9)
N(1)-P(1)-O(2)-C(7)	-64.7(9)
O(1)-P(1)-O(3)-C(8)	-55.2(9)
O(2)-P(1)-O(3)-C(8)	180.0(9)
N(1)-P(1)-O(3)-C(8)	68.9(9)
O(1)-P(1)-N(1)-C(1)	-178.4(9)
O(2)-P(1)-N(1)-C(1)	-49.2(9)
O(3)-P(1)-N(1)-C(1)	53.6(9)
P(1)-N(1)-C(1)-C(2)	-0.8(9)
P(1)-N(1)-C(1)-C(6)	179.0(9)
O(4)-N(2)-C(4)-C(3)	-5.0(9)
O(4)-N(2)-C(4)-C(5)	176.4(9)
O(5)-N(2)-C(4)-C(3)	172.9(9)
O(5)-N(2)-C(4)-C(5)	-5.8(9)
N(1)-C(1)-C(2)-C(3)	180.0(9)
C(6)-C(1)-C(2)-C(3)	0.0(9)
N(1)-C(1)-C(6)-C(5)	-179.0(9)
C(2)-C(1)-C(6)-C(5)	0.8(9)
C(1)-C(2)-C(3)-C(4)	0.0(9)
C(2)-C(3)-C(4)-N(2)	180.0(9)
C(2)-C(3)-C(4)-C(5)	-0.9(9)
N(2)-C(4)-C(5)-C(6)	180.0(9)
C(3)-C(4)-C(5)-C(6)	1.5(9)
C(4)-C(5)-C(6)-C(1)	-1.4(9)

(1.405(9) Å vs. 1.414(4) Å). This value is shorter than the value of 1.413(3) Å associated with this bond in acetanilide.[45]

The phosphoramidate group is planar as indicated by the O(1) – P – N – C(1) torsion angle (-178.4(9)°). The P – N – C(1) bond angle is significantly larger (∿10°) than the value of 120° expected for a sp^2 hybridised nitrogen atom. This angle is also enlarged in the *p*-methoxy analogue and in acetanilide.[45] The enlargement of the P – N – C(1) angle is possibly the result of repulsion between the bulky methoxy groups attached to phosphorus and the hydrogen atom attached to C(2). The phenyl ring is staggered with respect to the $CH_3O - P - OCH_3$ group, further minimising steric interactions between the N-aromatic group and the two phosphate ester groups. The phosphoramidate group and the benzene ring are coplanar; the P(1) – N(1) -- C(1) – C(2) torsion angle is -0.8(9)°. The co-planarity of these two groups is indicative of enhanced π-interaction of the lone pair on nitrogen with the benzene ring. This interaction is also suggested by the short C(1) – N bond length in this compound.

The Benzene Ring Geometry

Of particular interest in this compound are the interactions of the *p*-nitro substituent with the benzene ring and hence with the phosphoramidate group. As discussed earlier, these interactions of the substituent with the benzene ring may be discussed in terms of the distortions of the ideal symmetry of the benzene ring on substitution[46-48]. In particular the *ipso* angle, α, is sensitive to the σ-electron withdrawing or releasing effects of the substituent. In addition, in certain cases α is also

sensitive to the degree of π interaction of the substituent with the ring.

In the title compound the *ipso* angle of the nitro group , α_{NO_2} (C(3) – C(4) -- C(5)), is larger than the average value of 122.1(1)° obtained for many derivatives of nitrobenzene.[46] The α_{NO2} angle in the title compound is 124.7(10)°; the corresponding value of this angle in *p*-dinitrobenzene is 123.4(1)°[58]. The value of α_{NO_2}, 124.7(10)°, is consistent with the strong σ electron withdrawing character of the nitro substituent[46]. Larger than average values of α_{NO_2} are found in compounds where the nitro group is *para* to a strong π donor and is attributed to the effects of through-conjugation[58,59]. In this compound both the nitro and phosphoramidate groups are co-planar with the benzene ring, favouring through-conjugation.

The value of the amide *ipso* angle, α_{NH} (C(6) – C(1) – C(2)), is 120.3(9)°which is similar (within experimental error) to the standard value of 119.2(2)°determined for many derivatives of aniline (Ph – NH – X)[50], and is also similar to the value of 120.0(2)° found in 3-pyridyl-carboxanilide[60]. Generally α_{NH} is reduced when the substituent (– NH – X) is π-electron releasing but deviations occur when through-conjugation occurs[50]. The internal angle *para* to the substituent, δ, is also sensitive to the nature of the substituent although much less so than the *ipso* angle, α. It has been found that δ is larger when the benzene ring is substituted with a strong π-acceptor[50]. In the benzene-diazonium cation δ has been estimated at 121.6° by *ab initio* MO calculations[61]. It is therefore expected that the nitro group in the title compound would have the effect of increasing α_{NH}.

The equation of the least-squares plane calculated through the six atoms defining the benzene ring is given in Table 5.9. The deviations of the ring atoms out of the mean plane show that there is only a slight deformation of the planarity of the benzene ring. The geometry of the nitro group, including the C(4) -- N(2) bond, is virtually identical to that observed in *p*-dinitrobenzene and *p*-nitrobenzoic acid[57,58]. The nitro group is slightly twisted out of the plane of the benzene ring.

Hydrogen Bonding and Crystal Packing

A perspective view of the hydrogen bonding scheme in the title compound is given in Figure 5.1 and the hydrogen bonding data given in Table 5.10. The hydrogen bonding in this compound is unusual. Although the phosphoramidate group is aligned in the *cis* orientation, dimers similar to those found in the *p*-methoxy analogue are not formed. Individual molecules participate in an intermolecular network of N – H ---- O = P hydrogen bonds extending parallel to the *b* axis. The hydrogen bond is classified as of moderate strength[54] and is similar to the average value of 2.876(4) Å found in the *p*-methoxy analogue. The hydrogen bond angle, N – H ---- O, is 164.4(9)°.

Generally *cis*-amides (carboxylic) form cyclic hydrogen bonded pairs and when free rotation of the amide bond permits both *cis* and *trans* conformations intermolecular N – H ---- O hydrogen bonds are associated with the *trans* conformation[33,62,63]. In certain *cis*-fused amides intermolecular hydrogen bonding similar to that found in the title compound has been observed. In the crystal structure of C(*L*-pro-*D*-phe),[64] glutarimide[65]

Table 5.9 The Least-Squares Plane Calculated through the Benzene Ring and the Deviations of Selected Atoms from the Plane with e.s.d.'s in Parentheses.

Equation of the plane:

$6.17x + 0.21y + 11.93z = 4.35$

Deviations (Å):

P(1)	0.04(1)
O(1)	0.08(1)
N(1)	0.01(1)
*C(1)	0.00(1)
*C(2)	0.00(1)
*C(3)	0.00(1)
*C(4)	-0.01(1)
*C(5)	0.01(1)
*C(6)	-0.01(1)
N(2)	0.01(1)
O(4)	-0.07(1)
O(5)	0.14(1)

*Atoms used to calculate the least-squares plane

Table 5.10 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen Bonding	
N(1)-H(1)	1.00(5) Å
H(1).....O(1) [1]	1.85(5) Å
N(1).....O(1) [1]	2.83(1) Å
N(1)-H(1)-O(1)	164.4(9)°
Close contacts	
C(6).....O(1) [1]	3.37(1)
C(5).....O(2) [2]	3.45(1)
C(6).....O(2) [2]	3.44(1)
O(4).....O(3) [4]	3.46(1)
O(5).....O(4) [3]	3.49(1)
C(2).....O(4) [4]	3.40(1)
C(8).....O(4) [4]	3.49(1)
C(6).....O(4) [5]	3.47(1)
O(5).....O(5) [3]	3.09(1)
N(2).....O(5) [3]	3.02(1)
C(4).....O(5) [3]	3.31(1)
C(5).....O(5) [5]	3.32(1)
C(7).....O(5) [7]	3.37(1)
N(2).....N(2) [3]	3.44(1)
C(4).....N(2) [6]	3.45(1)
C(5).....N(2) [6]	3.31(1)
C(3).....C(3) [6]	3.47(1)

Symmetry code:

[1] 0.5-x -0.5+y z	[4] 1-x -y -z
[2] x -1+y z	[5] 1-x y 0.5-z
[3] 1-x -1-y -z	[6] -x y 0.5-z
	[7] 1-x 1+y 0.5-z

and C(*L*-his-*L*-asp).$3H_2O$[66] hydrogen bonded molecules are related by twofold screw axes. The repeat distances along the direction of the chain in these crystals are close to each other, varying from 6.143 to 7.416 Å. In the title compound the repeat distance along the direction of the chain (*b*) is 7.421 Å.

Leiserowitz *et al.* have considered the crystal packing of amides in terms of Coulomb forces[67]. It is concluded that a full understanding why a given molecule adopts its actual hydrogen motif requires overall packing to be taken into consideration. Leiserowitz *et al.* illustrate however that in carboxylic amides the cyclic dimer has one significant advantage over the intermolecular motif: the dimers are freer to adopt a more satisfactory stack than the molecules related by a translation motif, which are constrained by the N — H ---- O bond to adopt a more fixed arrangement.

A projection of the crystal structure down the *b* axis is shown in Figure 5.2. The molecules pack in alternate layers slightly tilted to the *c* axis. The spacing between the layers is approximately 3.5 Å which is within the range found in aromatic compounds which pack in a layer motif[68]. Figure 5.3 shows how individual molecules in alternate layers are related. Molecules above and below each other are twisted so as to reduce overlap. The structure is characterised by a number of close contacts ($d < 3.5$ Å). These mainly involve interaction between the nitro group and the benzene ring of a symmetry related molecule (refer to Table 5.10).

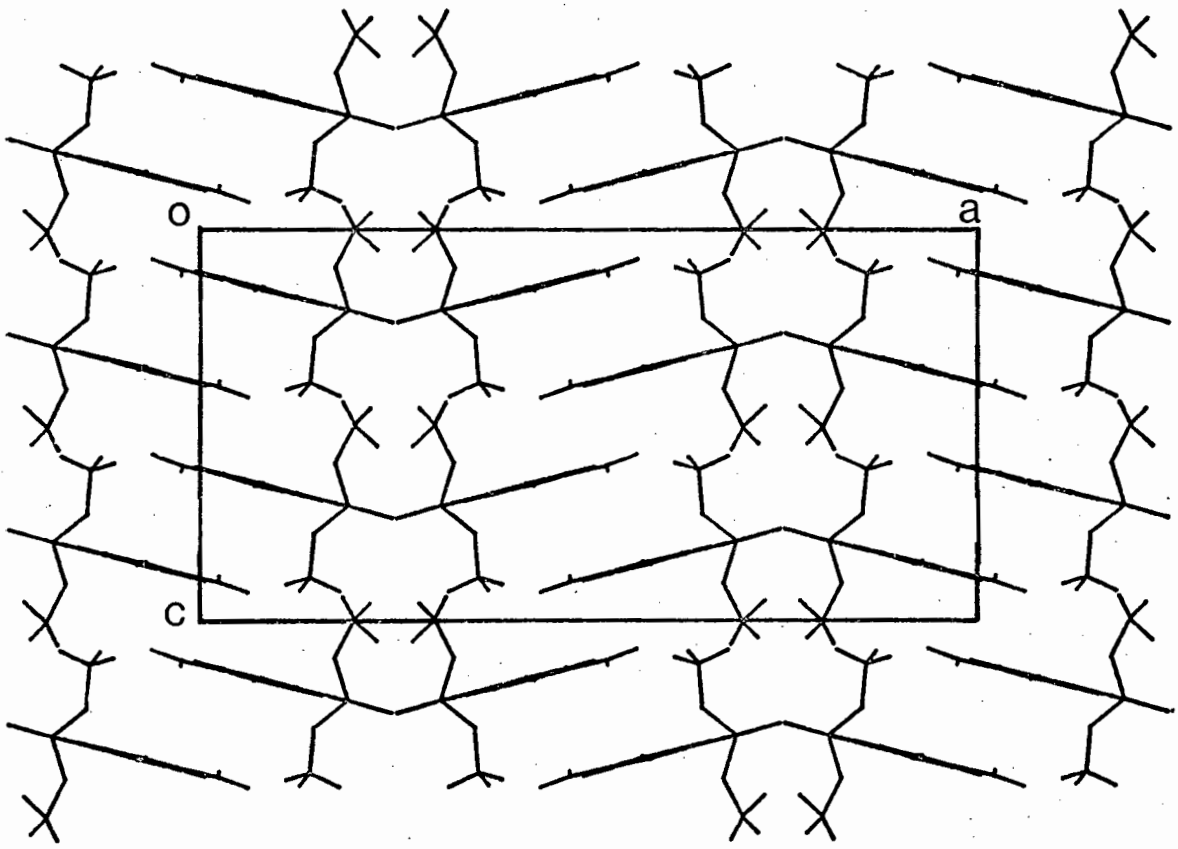


Figure 5.2 A projection of the crystal structure

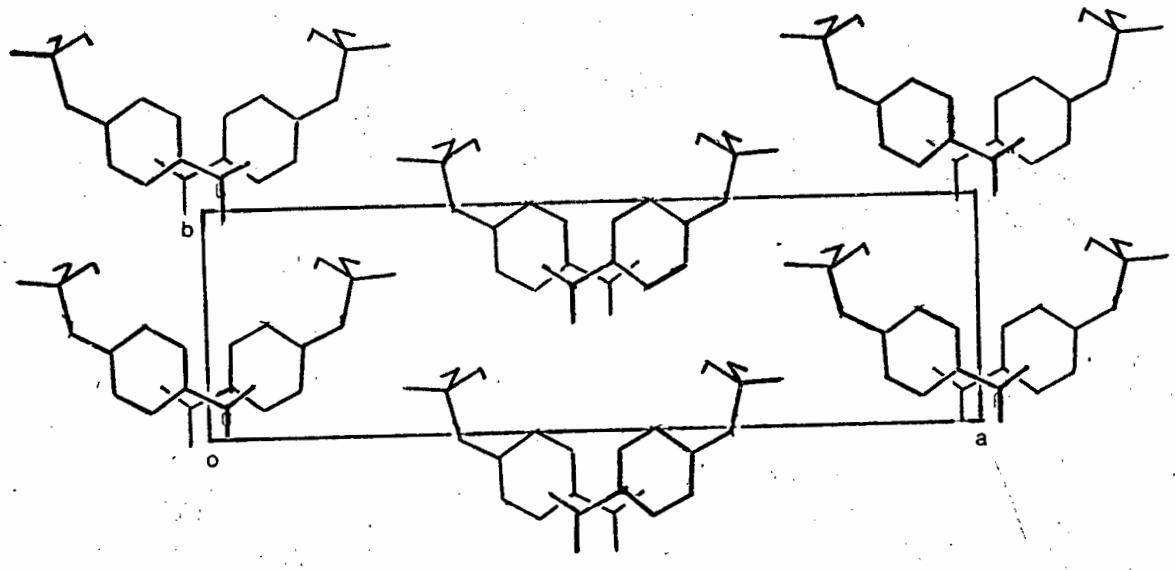


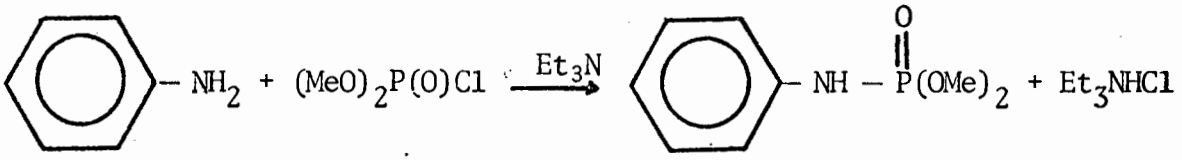
Figure 5.3 A view of the crystal layer structure

CHAPTER SIX

6. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIMETHYL-N-PHENYL-PHOSPHORAMIDATE

6.1 Experimental

The title compound was synthesized according to the reaction:[38]



The experimental procedure is similar to that described for the *p*-methoxy analogue in chapter 4 and will not be repeated. The crude product was recrystallized as colourless needles from petroleum ether (60-90°C). Found for $C_8H_{12}NO_3P$: C = 47.75 %, H = 5.95 %, N = 6.90 %. Calculated: C = 47.76 %, H = 6.01 %, N = 6.96 %. The density was determined by flotation in a calibrated density column.[39]

Preliminary oscillation and Weissenberg photography (*h*0*l*, *h*1*l*, *hk*0, *hk*1) indicated the monoclinic space group $P2_1/c$.[11] Inspection of the diffractometer data list revealed the alternative setting $P2_1/n$. This relationship between the space group $P2_1/c$ and $P2_1/n$ was shown in Chapter 4. The structure was subsequently solved and refined in the space group $P2_1/n$. The crystal details and the details of the diffractometer data collection are given in Table 6.1. No absorption corrections were required (refer to Table 6.1).

TABLE 6.1 Crystal Data and Experimental and Refinement Parameters for the Structure Analysis

Crystal Data	
Molecular formula	$C_8H_{12}NO_3P$
M_r	201.2
Space group	$P2_1/n$
a	16.929(8) Å
b	7.706(4) Å
c	15.883(8) Å
β	90.24(2)°
V	2072.0 Å^3
D_m	1.28 Mgm^{-3}
D_c	1.29 Mgm^{-3} for $Z = 8$
μ(Mo-K_α)	0.197 mm^{-1}
μR_{max}, μR_{min}	0.09, 0.03
A*	1.0
F(000)	824
Data Collection	
Crystal dimensions	0.32 x 0.68 x 0.96 mm
Scan mode	$\omega - 2\theta$
Scan width	1.5° θ
Scan speed	0.05° θ s^{-1}
Range scanned (2)	6-40°
Stability of standard reflections	1.0 %
Number of reflections collected	2803
Number of 'observed' reflections	2148 with $I_{rel} > 2\sigma I_{rel}$
Final Refinement	
Number of variables	259
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.064
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.074
Weighting scheme w	$(\sigma^2 F + 0.002F^2)^{-1}$
[a] U_{iso}(aromatic H)	0.10(1), 0.08(1) (Å^2)
[a] U_{iso}(methyl H)	0.20(2), 0.17(2) (Å^2)
[a] U_{iso}(amide H)	0.07(2), 0.10(2) (Å^2)

[a] values for molecules A and B respectively

6.2 Solution and Refinement of the Structure

The structure was solved by the automatic centrosymmetric direct methods routine of the SHELX[12] program system, in which the first *E*-map yielded the positions of all the heavy atoms. Most of the hydrogen atoms, including the amide hydrogen atoms, were located in a difference electron-density map calculated after a number of cycles of least-squares refinement. The structure crystallizes as two crystallographically independent molecules. In subsequent least-squares refinements all the heavy atoms were treated anisotropically, the methyl hydrogen atoms refined as rigid groups and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atoms were constrained at 1.00 ± 0.01 Å from the nitrogen atoms. The isotropic temperature factors of the methyl, aromatic and amide hydrogen atoms in each molecule were treated as three single parameters. In the final cycle of least-squares refinement the average shift to error ratio was less than 1.5 %. A final difference electron-density map calculated after the final least-squares refinement revealed no peaks of height >.30 e $Å^{-3}$. An analysis of variance calculated after the final least-squares refinement indicated that the refinement had converged successfully (refer to Table 6.2). Details of the solution and refinement of the structure are given in Table 6.1. Lists of observed and calculated structure factors are given in Appendix 6 and final atomic coordinates and isotropic temperature factors in Tables 6.3, 6.4 and 6.5 respectively.

Table 6.2 Analysis of Variance[a]

(a) By parity groups

Groups	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	329	223	255	260	216	329	265	271	2148
V	115	113	116	111	113	124	110	106	114

(b) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.19	.21	.23	.25	.28	.31	.34	.39	.46	1.00
N	290	190	202	177	248	225	177	229	201	209	
V	124	127	115	122	125	121	113	107	91	80	

(c) As a function of sin (θ)

sin(θ)	0	.17	.22	.25	.28	.30	.32	.34	.36	.38	.40
N	218	242	208	256	206	219	223	225	229	122	
V	140	117	109	100	117	101	120	111	111	108	

(d) As a function of |Miller index|

$\|h\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	89	181	182	180	167	167	159	149	136	136	121	104	97	85	195
V	139	124	124	131	126	128	116	120	105	97	104	89	87	92	87

$\|k\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	214	384	384	332	288	230	177	105	34	0	0	0	0	0	0
V	134	104	113	117	109	107	110	123	139	0	0	0	0	0	0

$\|l\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	98	194	197	199	181	185	165	158	135	139	123	104	88	62	120
V	115	115	120	118	114	104	101	109	104	103	105	112	122	148	137

[a] N = Number of reflections in the group; $V = 100[M(\Sigma w|F_o-F_c|^2)/N\Sigma w]$; M = Total number of reflections

Table 6.3 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	Molecule A			Molecule B		
	x	y	z	x	y	z
P(1)	-234(1)	3965(2)	1340(1)	5835(1)	2079(2)	481(1)
O(1)	-601(2)	3397(5)	555(2)	4986(2)	2204(5)	364(3)
O(2)	101(2)	2468(5)	1909(2)	6311(2)	3410(6)	-20(3)
O(3)	-797(2)	4889(5)	1977(2)	6152(3)	2478(7)	1412(3)
N(1)	462(2)	5357(5)	1147(2)	6150(2)	137(5)	340(3)
C(1)	1010(3)	6183(6)	1702(3)	6917(3)	-571(6)	421(3)
C(2)	966(3)	6043(7)	2571(3)	7581(3)	438(6)	548(3)
C(3)	1514(4)	6881(8)	3066(4)	8314(3)	-330(7)	616(3)
C(4)	2111(4)	7878(8)	2700(4)	8400(4)	-2127(8)	558(4)
C(5)	2149(3)	8017(8)	1838(4)	7737(4)	-3116(7)	427(3)
C(6)	1603(3)	7173(7)	1342(3)	6995(3)	-2369(6)	362(3)
C(7)	634(4)	1178(9)	1555(5)	6076(5)	3778(11)	-898(5)
C(8)	-1493(4)	4010(9)	2297(5)	5957(5)	3890(9)	1874(5)

Table 6.4 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms.

	Molecule A			Molecule B		
	x	y	z	x	y	z
H(1)	49	567	54	574	-69	13
H(21)	51	527	286	752	183	60
H(31)	147	680	374	883	47	72
H(41)	254	852	309	898	-272	62
H(51)	261	878	155	780	-451	38
H(61)	163	730	67	648	-317	26
H(71)	83	22	201	650	424	-135
H(72)	114	186	131	549	380	-118
H(73)	32	54	105	622	247	-72
H(81)	-174	490	275	630	422	243
H(82)	-125	289	262	534	391	202
H(83)	-195	359	187	608	482	138

Table 6.5 Anisotropic Temperature Factors ($Å^2 \times 10^3$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
	Molecule A					
P(1)	67(1)	51(1)	48(1)	3(1)	-2(1)	-5(1)
O(1)	86(3)	71(2)	51(2)	3(2)	-6(2)	-23(2)
O(2)	91(3)	56(2)	65(2)	10(2)	6(2)	11(2)
O(3)	70(2)	63(2)	70(2)	-7(2)	9(2)	0(2)
N(1)	72(3)	65(3)	44(2)	4(2)	-2(2)	-11(2)
C(1)	53(3)	49(3)	56(3)	-5(2)	-3(2)	7(2)
C(2)	74(3)	73(4)	51(3)	-7(3)	-7(3)	-1(3)
C(3)	81(4)	91(4)	63(3)	-18(3)	-13(3)	10(3)
C(4)	63(3)	84(4)	106(5)	-35(4)	-23(3)	16(3)
C(5)	64(3)	75(4)	91(5)	-22(3)	2(3)	-6(3)
C(6)	65(3)	59(3)	70(3)	-8(3)	3(3)	0(3)
C(7)	107(5)	81(5)	113(5)	6(4)	12(4)	28(4)
C(8)	96(5)	103(5)	99(5)	-16(4)	35(4)	-24(4)
	Molecule B					
P(1)	53(1)	47(1)	95(1)	-8(1)	5(1)	-2(1)
O(1)	53(2)	57(2)	126(3)	-15(2)	5(2)	2(2)
O(2)	75(3)	67(3)	160(5)	31(3)	-1(3)	-5(2)
O(3)	108(3)	123(4)	117(4)	-51(3)	-16(3)	25(3)
N(1)	51(2)	44(2)	105(3)	-7(2)	0(2)	-6(2)
C(1)	53(3)	48(3)	52(3)	5(2)	2(2)	-4(2)
C(2)	56(3)	48(3)	76(3)	2(2)	2(2)	-5(2)
C(3)	56(3)	67(4)	91(4)	9(3)	-9(3)	-6(3)
C(4)	69(4)	75(4)	100(4)	11(3)	-12(3)	14(3)
C(5)	87(4)	48(3)	103(4)	7(3)	-7(3)	8(3)
C(6)	68(3)	43(3)	73(3)	5(2)	-4(3)	1(3)
C(7)	121(6)	127(7)	101(6)	36(5)	15(5)	19(5)
C(8)	143(6)	94(5)	93(5)	-28(4)	6(4)	14(5)

6.3 Description of the Structure and Discussion

A perspective view of the title compound is given in Figure 6.1. Principal bond lengths, bond angles and torsion angles are given in Tables 6.6, 6.7 and 6.8 respectively.

Molecular Structure

The title compound crystallizes with two independent molecules (A and B) in the asymmetric unit. The two independent molecules form two dimers related by centres of symmetry via $-N-H\cdots O=P-$ hydrogen bonds. This is shown in Figure 6.2. The magnitudes of the torsion angles in Table 5.8 show that molecules A & B have similar conformations. Although the torsion angles have similar magnitudes in both molecules, some of the torsion angles have opposite signs. Particularly the torsion angles associated with the $(O)P(OR)_2N-$ group have opposite signs. These groups can be considered as pseudo mirror images of each other, since in terms of the definition of the torsion angle, torsion angles which are mirror images of each other have opposite signs.*

There are two possible ways in which dimerization can occur when there are two independent molecules in the asymmetric unit. If A and B are independent molecules then scheme (I) below represents the first case.

---

* The torsion angle was defined in Chapter 4. This definition implies that a positive value of a torsion angle (ψ) of a sequence of atoms ABCD forms a right handed screw. If ψ(ABCD) is positive, then so is ψ(DCBA) - a right-handed corkscrew remains right-handed when it is turned from back to front. Also in terms of this definition the torsion angle is undefined at $\psi = 0°$ and 180°.

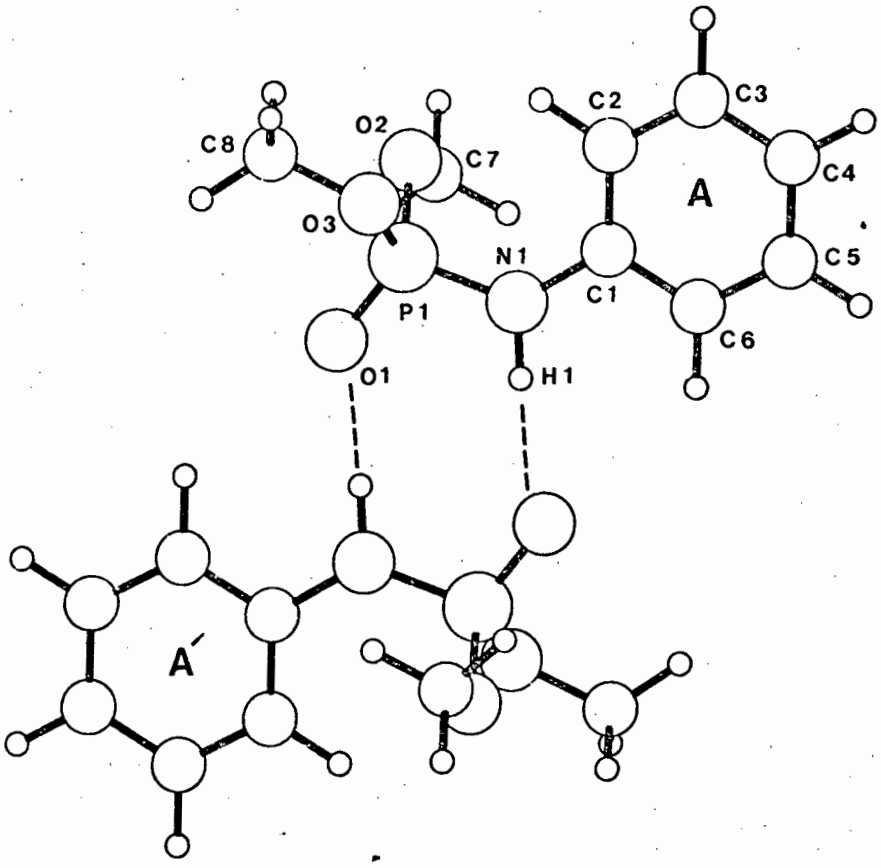


Figure 6.1 Perspective views of molecules A and B

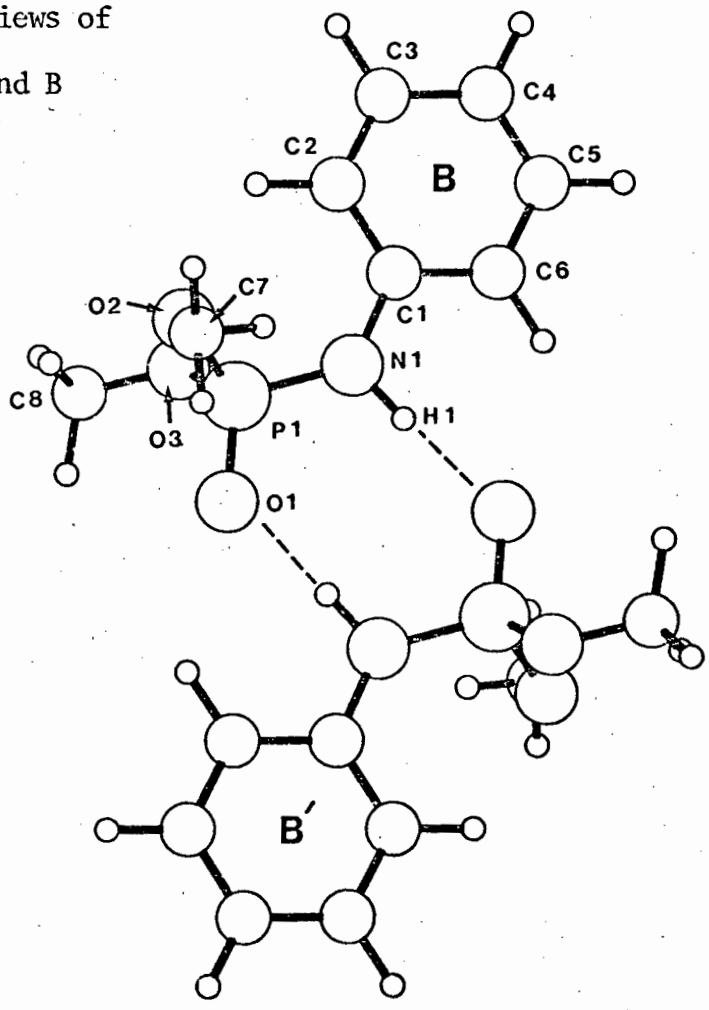


Table 6.6 Principal Bond Lengths (Å) with e.s.d.'s in Parentheses.

Bond	Molecule A	Molecule B
P(1)-O(1)	1.458(3)	1.453(4)
P(1)-O(2)	1.570(4)	1.529(5)
P(1)-O(3)	1.564(3)	1.601(5)
P(1)-N(1)	1.623(4)	1.604(4)
N(1)-C(1)	1.427(6)	1.415(6)
C(1)-C(2)	1.387(7)	1.379(6)
C(2)-C(3)	1.375(7)	1.380(7)
C(3)-C(4)	1.399(9)	1.395(8)
C(4)-C(5)	1.375(8)	1.373(8)
C(5)-C(6)	1.375(7)	1.384(7)
C(7)-C(1)	1.387(7)	1.395(6)
O(2)-C(7)	1.457(7)	1.476(8)
O(3)-C(8)	1.453(7)	1.353(7)

Table 6.7 Principal Bond Angles (°) with e.s.d.'s in Parentheses.

Angle	Molecule A	Molecule B
O(1)-P(1)-O(2)	115.0(2)	114.4(2)
O(1)-P(1)-O(3)	115.5(2)	115.6(2)
O(1)-P(1)-N(1)	110.1(2)	111.9(2)
O(2)-P(1)-N(1)	109.5(2)	112.2(3)
O(2)-P(1)-O(3)	100.5(2)	100.2(3)
O(3)-P(1)-N(1)	105.4(2)	101.4(3)
P(1)-N(1)-C(1)	130.5(3)	130.7(3)
C(6)-C(1)-C(2)	119.7(5)	119.5(5)
C(1)-C(2)-C(3)	119.6(5)	120.1(5)
C(2)-C(3)-C(4)	120.5(5)	121.0(5)
C(3)-C(4)-C(5)	119.6(5)	118.4(5)
C(4)-C(5)-C(6)	119.9(6)	121.4(5)
C(5)-C(6)-C(1)	120.7(5)	119.6(5)
P(1)-O(2)-C(7)	120.1(4)	118.7(4)
P(1)-O(3)-C(8)	120.8(4)	125.0(5)
C(6)-C(1)-N(1)	117.5(4)	117.6(4)
C(2)-C(1)-N(1)	122.8(4)	122.6(4)

Table 6.8 Selected Torsion Angles (°) with e.s.d.'s in Parentheses.

Torsion angle	Molecule A	Molecule B
O(1)-P(1)-O(2)-C(7)	51.8(5)	-42.0(5)
O(3)-P(1)-O(2)-C(7)	176.5(5)	-166.3(5)
N(1)-P(1)-O(2)-C(7)	-72.8(5)	86.8(5)
O(1)-P(1)-O(3)-C(8)	58.6(5)	-49.9(5)
O(2)-P(1)-O(3)-C(8)	-65.9(5)	73.6(5)
N(1)-P(1)-O(3)-C(8)	180.0(5)	-171.2(5)
O(1)-P(1)-N(1)-C(1)	-175.8(5)	-177.3(5)
O(2)-P(1)-N(1)-C(1)	-48.4(5)	52.7(5)
O(3)-P(1)-N(1)-C(1)	58.9(5)	-53.4(5)
P(1)-N(1)-C(1)-C(2)	-7.9(5)	-8.7(5)
P(1)-N(1)-C(1)-C(6)	172.7(5)	171.8(5)
N(1)-C(1)-C(2)-C(3)	180.0(5)	180.0(5)
C(6)-C(1)-C(2)-C(3)	0.0(5)	0.0(5)
N(1)-C(1)-C(6)-C(5)	180.0(5)	179.2(5)
C(2)-C(1)-C(6)-C(5)	0.0(5)	0.0(5)
C(1)-C(2)-C(3)-C(4)	0.0(5)	0.0(5)
C(2)-C(3)-C(4)-C(5)	0.0(5)	0.0(5)
C(3)-C(4)-C(5)-C(6)	0.0(5)	0.0(5)
C(4)-C(5)-C(6)-C(1)	0.0(5)	0.0(5)

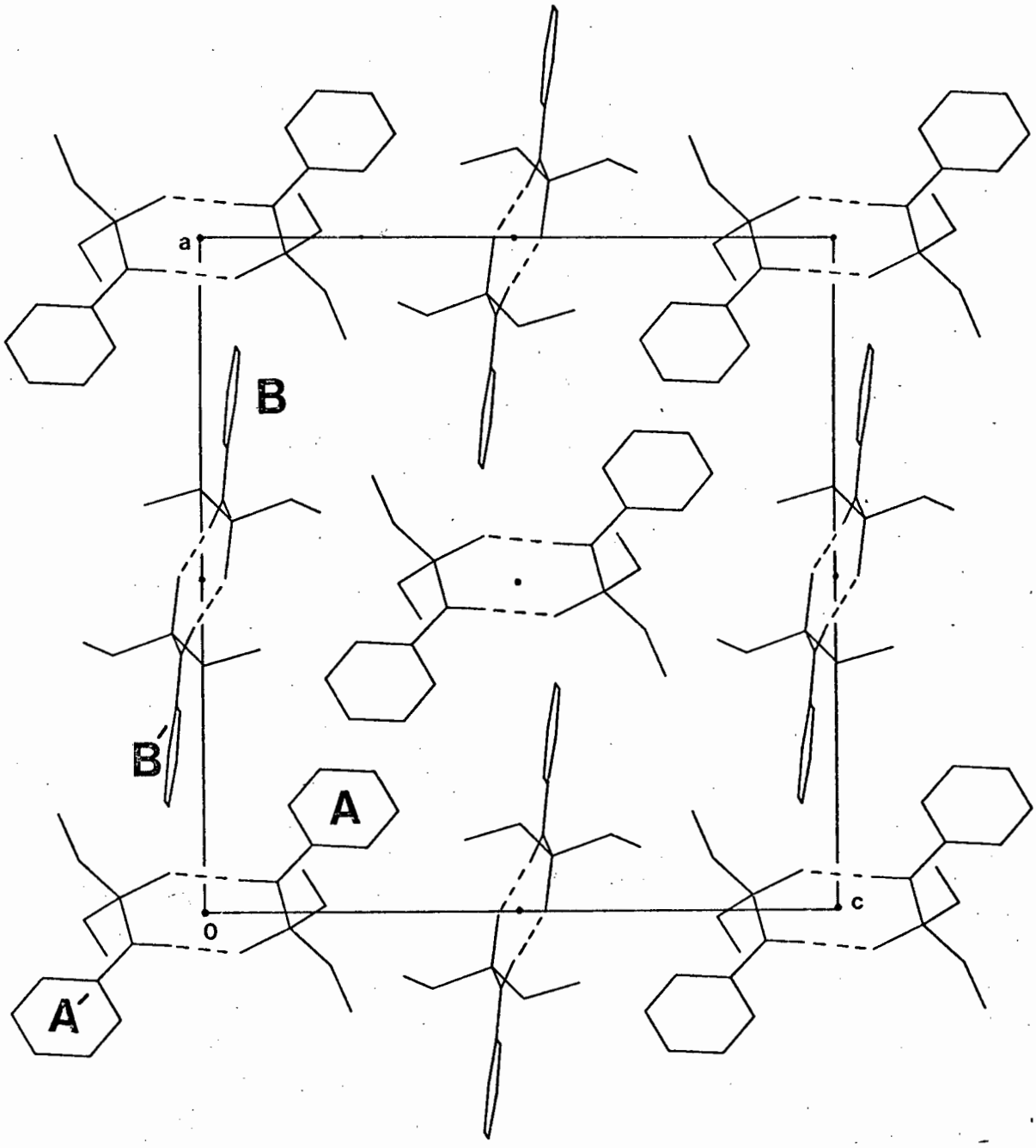
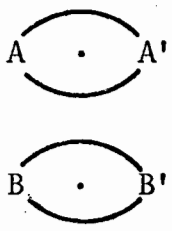


Figure 6.2 A projection of the crystal packing

In this case the dimer is formed between the two independent molecules and translation through the centre of symmetry generates a dimer of opposite chirality. This situation occurs in the *p*-methoxy analogue. The second case occurs in the title compound and is shown in scheme (II).

Scheme (I)



Scheme (II)

In this case the two independent molecules form independent dimers by translation through the centres of symmetry. This leads to two symmetric dimers. In the first case the asymmetric unit was a dimeric species. In the second case the asymmetric unit consists of the molecules A and B which do not associate with each other in dimer formation.

The coordination of the phosphorus atom is similar to that observed in the *p*-methoxy and *p*-nitro analogues. Once again the O(2) – P – O(3) angle is compressed while the O(1) -- P – O /N angles are enlarged. The values of the P – O(1) bond lengths (1.458(3) Å and 1.453(4) Å) lie within the range of values observed in the previous two structures. The P – N bond lengths for molecules A and B are significantly different (in terms of the estimated standard deviations calculated by SHELX) from each other and the average value of 1.613(4) Å is shorter than that observed in the two previous structures. The N-C(1) bond distances, 1.427(6) Å and 1.415(6) Å with mean 1.421(6) Å, are similar (within experimental error) to the value of 1.413(3) Å in the analogous acetanilide.[45] The phosphoramidate group,

is planar within experimental error. The benzene ring is also co-planar with the phosphoramidate group (the P(1) – N(1) – C(1) – C(2) torsion angles are -7.9(5)° and -8.7(5)° respectively).

The Benzene Ring Geometry

As might be expected the distortions of the ideal symmetry of the benzene ring observed in the previous two *p*-substituted compounds are not observed in the title compound. In the title compound the *ipso* angles of the amide group, C(6) – C(1) – C(2), are 119.7(5)° and 119.5(5)°. These values are virtually identical with the 'standard' value of 119.1(2)° determined for the series of aniline derivatives.[50]

There are no discernable distortions of the ideal symmetry of the benzene ring. The internal torsion angles of the benzene ring are all zero and all the deviations of the ring atoms from the least-squares plane calculated through the benzene ring are within the estimated standard deviations (Table 6.9). The amide group therefore has only a small effect on the π-electron system of the benzene ring.

Hydrogen Bonding and Crystal Packing

A perspective view of the hydrogen bonding scheme in the title compound is given in Figure 6.2 and details of the hydrogen bonding in Table 6.10. There are two independent N – H ---- O = P hydrogen bonds. Each dimer lies on a centre of symmetry. The hydrogen bond distances, N ---- O,

Table 6.9 The Least-Squares Planes Calculated through the Benzene Rings with the Deviations of Selected Atoms from the Planes with e.s.d.'s in Parentheses.

Equations of the planes:

Molecule A: $-10.028x + 6.204y + 0.530z = 2.915$

Molecule B: $-2.103x - 0.625y + 15.715z = -0.759$

Deviations (Å) :

	Molecule A	Molecule B
N(1)	0.006(8)	0.009(8)
*C(1)	-0.001(8)	0.002(8)
*C(2)	0.002(8)	-0.001(8)
*C(3)	-0.001(8)	-0.001(8)
*C(4)	-0.007(8)	0.003(8)
*C(5)	0.002(8)	-0.002(8)
*C(6)	0.001(8)	0.005(8)

*Atoms used in the calculation of the least-squares plane

Table 6.10 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen bonding	Molecule A	Molecule B
N(1)-H(1)	1.00(1) Å	1.00(1) Å
H(1).....O(1)	1.89(1) Å [1]	1.87(1) Å [2]
N(1).....O(1)	2.88(1) Å [1]	2.86(1) Å [2]
N(1)-H(1)-O(1)	171.1(9) °	174.6(9) °
Close contacts		
C(3B).....O(1A) [3]	3.410(8)	
C(6A).....O(1A) [1]	3.479(8)	
C(4B).....O(3A) [4]	3.492(8)	
O(3B).....O(3A) [5]	3.302(8)	
O(3B).....C(8A) [3]	3.416(8)	

Symmetry codes:

[1] -x 1-y -z

[2] 1-x -y -z

[3] 1+x y z

[4] 1+x -1+y z

[5] 0.5-x -0.5+y 0.5-z

are similar to the values of 2.877(4) Å and 2.886(4) Å observed in the *p*-methoxy analogue which is also dimerized. In the latter compound however, there are two independent hydrogen bonds per dimer; in the title compound there is only one hydrogen bond length per dimer. In the title compound the hydrogen bond angles are 171.1(9)° and 174.6(9)°.

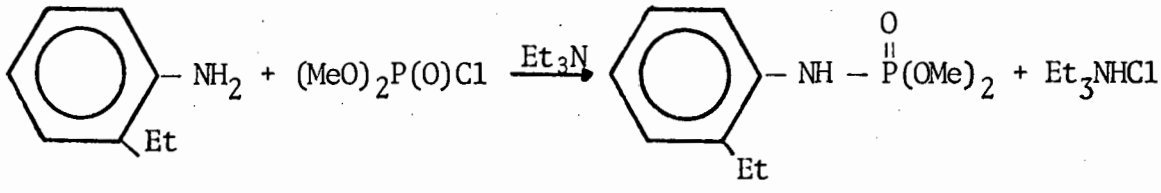
The benzene rings of symmetrically related molecules pack in layers with layers of type A molecules perpendicular to layers of type B molecules. Type A molecules stack parallel to the *b* axis and type B molecules stack parallel to the *a* axis. Close contacts (<3.5 Å) are listed in Table 6.10.

CHAPTER SEVEN

7. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIMETHYL-N-(2-ETHYLPHENYL)-PHOSPHORAMIDATE

7.1 Experimental

The title compound was synthesized according to the reaction below. The reaction procedure is the same as that for the *p*-methoxy analogue and was described in chapter 4.[38]



The product was crystallised from the resulting oil on standing and washed with a small quantity of petroleum ether (60-90°C). These crystals had a badly defined morphology. Hence some of the material was recrystallized from petroleum ether (60-90°C). Small well-defined colourless needles were produced. During preliminary photography it was found that crystals from the two crystallizations revealed different cell parameters. Oscillation and Weissenberg photography (*hk*0, *hk*1, 0*kl* and 1*kl*) revealed the monoclinic space group $P2_1/c^{11}$ in both cases with eight molecules per unit cell for the crystals obtained from the oil and four molecules per unit cell for the crystals obtained from the petroleum ether solution. A density determination using a calibrated density column revealed the same density for both kinds of crystals.[39] Diffractometer data were collected for both kinds of crystal and labelled structure (I) (from the oil) and structure (II) (from the petroleum ether). The crystal details as well as the details of the diffractometer data collection

are given in Table 7.1. There does not appear to be a direct relationship between the two structures. Once again no absorption correction was required (refer to Table 7.1).

7.2 Solution and Refinement of the Structures

Both structures were solved by the automatic centrosymmetric direct methods routine of the SHELX[12] program system in which an *E*-map revealed the positions of all the heavy atoms. Most of the hydrogen atoms were subsequently located in difference electron-density maps calculated after a number of cycles of least-squares refinement. In the final least-squares refinements both structures were treated in the same way, with all the heavy atoms treated anisotropically. The methyl and methylene hydrogen atoms were refined as rigid groups and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atoms were constrained at 1.00 ± 0.01 Å from their nitrogen atoms. The isotropic temperature factors of the methyl,methylene aromatic and amide hydrogen atoms were treated as four independent parameters in each independent molecule. In the final cycle of least-squares refinement the average shift to error ratio in both structures was less than 1 %. A final difference electron-density map calculated after the final cycle of least-squares refinement revealed no peaks of height > 0.1 e Å^{-3} in both cases. Tables 7.2 and 7.3 show the analyses of variance calculated after the final refinements. Details of the solution and refinement of the structures are given in Table 7.1, lists of observed and calculated structure factors in Appendix 7, final

Table 7.1 Crystal Data and Experimental and Refinement Parameters for the Structure Analyses

Crystal Data	Structure (I)	Structure (II)
Molecular formula	$C_{10}H_{16}NO_3P$	$C_{10}H_{16}NO_3P$
M_r	229.2	229.2
Space group	$P2_1/c$	$P2_1/c$
a (Å)	10.878(5)	7.071(4)
b (Å)	8.890(4)	17.059(9)
c (Å)	25.11(1)	10.221(5)
β (°)	92.18(2)	97.07(2)
V (Å^3)	2426.6	1223.5
D_m (Mgm^{-1})	1.23	1.23
D_c (Mgm^{-1})	1.25 (for $Z = 8$)	1.24 (for $Z = 4$)
$\mu(MoK_\alpha)$ (mm^{-1})	0.170	0.170
μR_{min}, μR_{max}	0.11, 0.05	0.07, 0.03
A*	1.1, 1.0	1.0
F(000)	976	488
Data Collection		
Crystal dimensions (mm)	1.3 x 0.9 x 0.6	0.32 x 0.8 x 0.8
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$
Scan width (°θ)	1.3	0.9
Scan speed (°θs^{-1})	0.04	0.03
Range scanned (2θ)(°)	6 - 46	6 - 44
Stability of standard reflections (%)	<1 %	<1 %
Number of reflections collected	3283	1609
Number of 'observed' reflections with $I_{rel} > 2\sigma I_{rel}$	2507	744
Final Refinement		
Number of variables	303	152
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.064	0.068
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.061	0.050
Weighting scheme w	$(\sigma^2 F)^{-1}$	$(\sigma^2 F)^{-1}$
U_{iso} (aromatic H) (Å^2)	0.114(9),0.118(9)	0.11(2)
U_{iso} (methyl H) (Å^2)	0.20(1),0.23(1)	0.22(2)
U_{iso} (methylene H) (Å^2)	0.11(1),0.18(2)	0.14(3)
U_{iso} (amide H) (Å^2)	0.07(1),0.09(2)	0.04(2)

Table 7.2 Analysis of variance for Structure (I)[a]

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	358	361	316	312	272	268	312	308	2507
V	124	125	122	128	103	103	127	104	118

(b) As a function of sin (θ)

sin(θ)	0	.17	.22	.25	.28	.30	.32	.34	.36	.37	.40
N	258	296	245	297	245	256	262	258	143	247	
V	185	148	118	109	126	94	99	83	72	80	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.17	.20	.22	.24	.27	.30	.34	.39	.48	1.00
N	255	354	220	207	265	228	257	230	265	226	
V	72	77	87	114	109	122	121	122	165	169	

(d) As a function of |Miller index|

$\lvert h \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	161	328	313	295	298	269	247	202	156	122	83	33	0	0	0
V	116	130	131	131	147	115	100	98	88	80	70	65	0	0	0

$\lvert k \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	212	402	391	362	325	276	235	164	96	44	0	0	0	0	0
V	135	139	132	141	104	87	92	79	87	63	0	0	0	0	0

$\lvert l \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	66	138	156	124	144	140	148	129	139	120	126	97	127	105	748
V	154	117	141	125	149	111	111	98	128	104	115	104	115	96	113

[a] N = Number of reflections in the group; $V = 100[M(\Sigma w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 7.3 Analysis of Variance for Structure (II)[a]

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	117	113	76	79	82	78	95	104	744
V	103	106	113	125	130	107	104	103	111

(b) As a function of sin (θ)

sin(θ)	0	.15	.19	.22	.25	.27	.29	.31	.32	.35	.40
N	78	77	78	94	76	73	78	41	92	57	
V	117	127	103	104	100	106	90	109	99	157	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.25	.27	.28	.30	.32	.35	.37	.42	.50	1.00
N	91	100	43	85	59	86	58	76	76	70	
V	135	115	105	92	96	102	98	103	119	124	

(d) As a function of |Miller index|

$\lvert h\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	90	176	153	126	108	71	19	1	0	0	0	0	0	0	0
V	126	118	114	102	90	101	132	179	0	0	0	0	0	0	0

$\lvert k\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	41	66	76	62	64	59	53	46	63	53	45	36	30	22	28
V	78	92	107	108	118	109	122	129	109	105	127	119	93	110	134

$\lvert l\rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	51	97	118	98	99	85	68	58	39	20	10	1	0	0	0
V	111	116	110	112	109	99	108	107	120	136	116	29	0	0	0

[a] N = Number of reflections in the group; $V = 100[M(\Sigma w|F_o-F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 7.4 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms of Structure (I) with e.s.d.'s in Parentheses.

	Molecule A			Molecule B		
	x	y	z	x	y	z
P(1)	3282(1)	998(1)	839(1)	6144(1)	-969(2)	1963(1)
O(1)	4524(3)	1460(3)	1005(1)	5258(3)	-1680(3)	1585(1)
O(2)	2217(3)	1876(3)	1095(1)	5743(3)	-941(4)	2557(1)
O(3)	2895(3)	1352(4)	245(1)	7361(3)	-1864(4)	2078(1)
N(1)	3089(3)	-796(4)	943(1)	6504(3)	696(4)	1757(1)
C(1)	2045(4)	-1699(5)	812(2)	7443(4)	1670(5)	1970(2)
C(2)	2119(4)	-3267(5)	865(2)	7982(5)	2706(7)	1651(2)
C(3)	1076(4)	-4120(6)	728(2)	8897(5)	3658(6)	1868(2)
C(4)	3(5)	-3460(7)	538(2)	9268(4)	3566(6)	2392(2)
C(5)	-68(4)	-1932(7)	489(2)	8729(4)	2547(7)	2712(2)
C(6)	951(4)	-1044(6)	630(2)	7814(4)	1592(6)	2506(2)
C(7)	2088(6)	1807(8)	1660(2)	4623(6)	-262(8)	2703(3)
C(8)	3661(6)	1282(10)	-164(3)	8085(6)	-2237(9)	1641(3)
C(9)	3271(5)	-4077(6)	1052(2)	7492(7)	3052(10)	1049(3)
C(10)	4094(6)	-4394(8)	599(3)	8269(9)	2192(11)	776(3)

Table 7.5 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms for Structure (II) with e.s.d.'s in Parentheses.

	x	y	z
P(1)	1818(4)	427(2)	1807(3)
O(1)	619(8)	820(3)	744(5)
O(2)	4011(11)	514(4)	1775(7)
O(3)	1656(10)	757(3)	3229(5)
N(1)	1372(9)	-509(4)	1846(6)
C(1)	1966(13)	-1067(6)	2851(7)
C(2)	978(14)	-1761(6)	2888(8)
C(3)	1577(15)	-2295(5)	3893(9)
C(4)	3127(17)	-2139(6)	4815(9)
C(5)	4068(14)	-1449(7)	4768(8)
C(6)	3543(13)	-908(6)	3774(9)
C(7)	4955(21)	990(11)	1190(13)
C(8)	-192(15)	783(6)	3667(9)
C(9)	-807(15)	-1954(5)	1941(9)
C(10)	-2479(15)	-1658(9)	2426(12)

Table 7.6 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms of Structure (I) .

	Molecule A			Molecule B		
	x	y	z	x	y	z
H(1)	382	-126	112	597	120	149
H(31)	111	-533	77	932	448	162
H(41)	-78	-415	43	998	430	255
H(51)	-91	-141	34	901	248	313
H(61)	89	17	60	739	79	276
H(71)	128	245	176	438	-3	311
H(72)	288	225	188	385	-76	249
H(73)	195	64	177	488	77	251
H(81)	335	176	-54	888	-292	175
H(82)	347	9	-16	837	-131	140
H(83)	464	146	-11	742	-292	142
H(91)	302	-513	123	758	423	95
H(92)	376	-339	134	655	270	98
H(101)	494	-494	73	771	292	52
H(102)	431	-334	41	801	104	71
H(103)	361	-511	32	923	234	69

Table 7.7 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms of Structure (II)

	x	y	z
H(1)	55	-72	106
H(31)	81	-284	395
H(41)	359	-257	557
H(51)	524	-132	551
H(61)	434	-37	372
H(71)	646	87	121
H(72)	474	135	203
H(73)	445	130	30
H(81)	20	100	466
H(82)	-107	27	370
H(83)	-98	123	308
H(91)	-91	-258	183
H(92)	-69	-169	100
H(101)	-323	-209	178
H(102)	-305	-108	219
H(103)	-264	-180	344

Table 7.8 Anisotropic Temperature Factors ($\AA^2 \times 10^3$) of the Heavy Atoms of Structure (I) with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
	Molecule A					
P(1)	58(1)	59(1)	67(1)	9(1)	-25(1)	-4(1)
O(1)	66(2)	66(2)	112(3)	20(2)	-43(2)	-17(2)
O(2)	81(2)	60(2)	88(3)	-4(2)	-17(2)	10(2)
O(3)	78(2)	125(3)	67(2)	32(2)	-30(2)	-23(2)
N(1)	52(2)	49(2)	79(3)	1(2)	-23(2)	-1(2)
C(1)	51(3)	58(3)	49(3)	-3(2)	-7(2)	-4(2)
C(2)	67(3)	56(3)	48(3)	-6(2)	-5(2)	-7(3)
C(3)	79(3)	70(3)	68(3)	-5(3)	-2(3)	-19(3)
C(4)	71(4)	94(5)	87(4)	-9(3)	-7(3)	-33(3)
C(5)	54(3)	99(5)	94(4)	5(4)	-12(3)	-11(3)
C(6)	51(3)	68(3)	83(3)	2(3)	-15(2)	-5(3)
C(7)	150(6)	117(5)	94(5)	-21(4)	11(4)	23(5)
C(8)	95(5)	293(12)	88(5)	45(6)	-5(4)	-40(6)
C(9)	82(4)	53(3)	106(4)	3(3)	-24(3)	-7(3)
C(10)	110(5)	115(6)	202(8)	17(6)	44(5)	32(5)
	Molecule B					
P(1)	64(1)	66(1)	79(1)	9(1)	-28(1)	-2(1)
O(1)	83(2)	63(2)	114(3)	8(2)	-54(2)	-13(2)
O(2)	90(3)	98(3)	89(3)	12(2)	-1(2)	-18(2)
O(3)	80(2)	82(2)	87(3)	6(2)	-36(2)	13(2)
N(1)	66(2)	61(3)	73(3)	14(2)	-32(2)	-10(2)
C(1)	59(3)	64(3)	54(3)	6(3)	-12(2)	-3(3)
C(2)	91(4)	109(5)	50(3)	22(3)	-23(3)	-23(4)
C(3)	90(4)	102(5)	89(4)	24(4)	-18(3)	-40(4)
C(4)	75(3)	91(4)	80(4)	-5(3)	-16(3)	-20(3)
C(5)	72(3)	105(4)	63(3)	-10(3)	-12(3)	-18(3)
C(6)	68(3)	87(4)	60(3)	5(3)	-12(3)	-10(3)
C(7)	81(5)	152(7)	155(6)	-50(5)	29(4)	-15(4)
C(8)	94(5)	167(7)	115(6)	-39(5)	-20(4)	32(5)
C(9)	116(6)	135(7)	165(8)	-31(6)	29(5)	-33(5)
C(10)	175(8)	160(8)	153(7)	-18(7)	-13(7)	-38(7)

Table 7.9 Anisotropic Temperature Factors ($\mathring{A}^2 \times 10^3$) of the Heavy Atoms of Structure (II) with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
P(1)	87(2)	64(2)	56(2)	2(2)	0(1)	-8(2)
O(1)	118(5)	65(4)	50(3)	7(3)	-13(4)	-2(4)
O(2)	87(6)	137(6)	121(6)	39(5)	4(4)	-32(5)
O(3)	115(6)	70(5)	59(4)	-10(3)	-12(4)	-5(4)
N(1)	80(5)	54(5)	43(4)	-4(4)	-2(4)	-12(5)
C(1)	61(7)	63(7)	41(6)	-1(5)	11(5)	12(6)
C(2)	82(8)	63(7)	46(6)	-6(6)	12(5)	2(6)
C(3)	123(9)	57(7)	58(6)	13(6)	12(6)	11(7)
C(4)	128(11)	71(9)	73(7)	15(7)	5(7)	43(8)
C(5)	85(8)	109(9)	59(7)	16(7)	-13(6)	32(8)
C(6)	63(7)	99(8)	62(6)	8(6)	-1(5)	2(6)
C(7)	137(13)	631(40)	183(14)	242(20)	-2(11)	-93(20)
C(8)	137(11)	114(10)	78(8)	2(6)	47(8)	25(8)
C(9)	99(9)	94(8)	66(7)	-1(6)	1(7)	-36(7)
C(10)	70(9)	313(19)	112(9)	-54(11)	6(8)	5(11)

atomic coordinates in Tables 7.4, 7.5, 7.6 and 7.7 and lists of anisotropic temperature factors in Tables 7.8 and 7.9 respectively.

7.3 Description of the Structures and Discussion

Perspective views of both structures are shown in Figures 7.1 and 7.2. Principal bond lengths, angles and torsion angles are listed in Tables 7.10 through 7.12 inclusive.

Molecular Structure

The title compound crystallizes in two distinct ways. In structure (I) the title compound crystallizes as a dimer with two independent molecules in the asymmetric unit. The two independent molecules are associated by two N — H ---- O = P hydrogen bonds. This situation was also observed in the *p*-methoxy analogue. However in structure (II) the title compound crystallizes with four molecules per unit cell. Centrosymmetrically related molecules form a dimer via two symmetrically-equivalent N — H ---- O = P hydrogen bonds.

In both structures the *cis* orientation of the phosphoramidate group is achieved despite the presence of the bulky *o*-ethyl group. The bulky methoxy groups attached to the phosphorus atom are oriented *trans* to the *o*-ethyl group and the N — H group is on the same side of the molecule as the *o*-ethyl group. The torsion angles listed in Table 7.12 indicate that both structures have similar conformations.

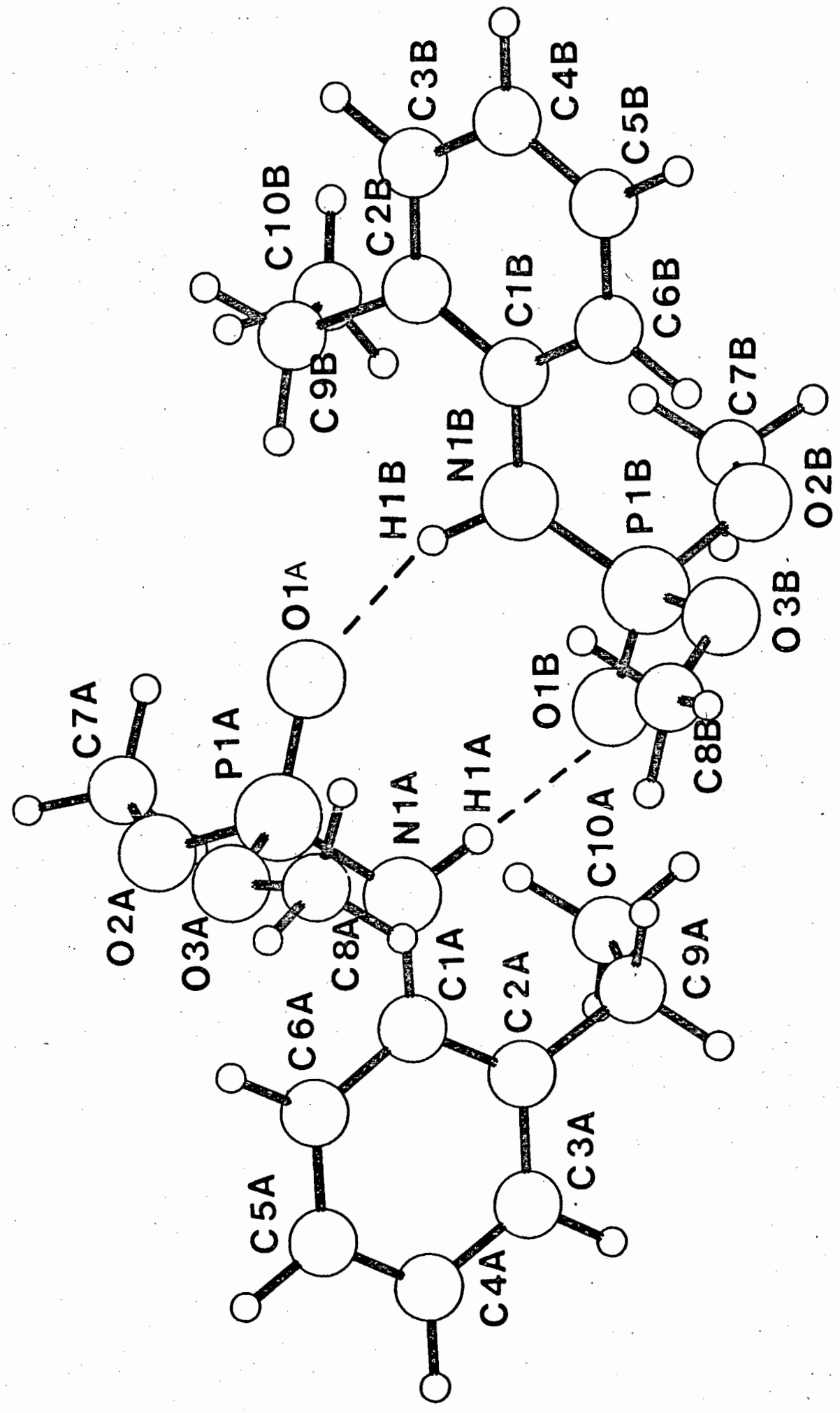


Figure 7.1 A perspective view of structure (I)

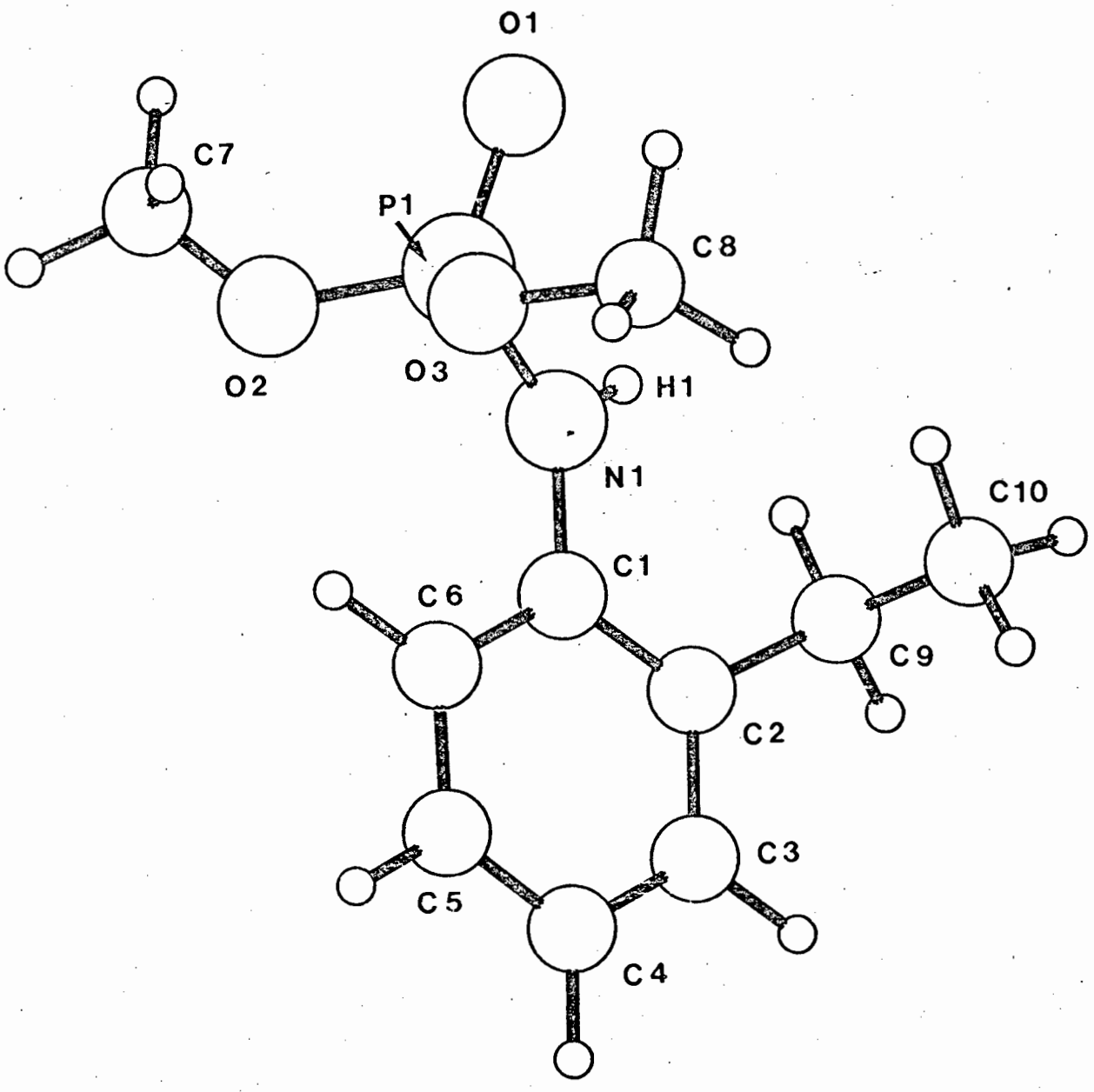


Figure 7.2 A perspective view of structure (II)

Table 7.10 Principal Bond Lengths (Å) of both Structures with e.s.d.'s in Parentheses.

Bond	Structure (I)		Structure (II)
	Molecule A	Molecule B	
P(1)-O(1)	1.458(3)	1.469(3)	1.456(5)
P(1)-O(2)	1.555(3)	1.569(4)	1.562(7)
P(1)-O(3)	1.567(3)	1.562(3)	1.576(6)
P(1)-N(1)	1.631(4)	1.622(4)	1.629(6)
C(7)-O(2)	1.434(6)	1.421(6)	1.249(11)
C(8)-O(3)	1.348(6)	1.415(6)	1.434(9)
C(1)-N(1)	1.419(5)	1.427(5)	1.425(9)
C(2)-C(1)	1.402(5)	1.368(6)	1.378(10)
C(6)-C(1)	1.387(5)	1.391(5)	1.396(10)
C(3)-C(2)	1.397(6)	1.401(6)	1.399(10)
C(9)-C(2)	1.505(6)	1.614(9)	1.529(10)
C(4)-C(3)	1.376(6)	1.363(6)	1.381(11)
C(5)-C(4)	1.365(7)	1.359(6)	1.356(11)
C(6)-C(5)	1.395(6)	1.393(6)	1.389(10)
C(10)-C(9)	1.502(7)	1.346(9)	1.429(10)

Table 7.11 Principal Bond Angles (°) of both Structures with e.s.d.'s in Parentheses.

Angle	Structure (I)		Structure (II)
	Molecule A	Molecule B	
O(2)-P(1)-O(1)	116.0(2)	114.9(2)	115.4(4)
O(3)-P(1)-O(1)	115.3(2)	115.5(2)	114.9(3)
O(3)-P(1)-O(2)	96.6(2)	95.6(2)	99.8(4)
N(1)-P(1)-O(1)	110.7(2)	110.3(2)	111.9(4)
N(1)-P(1)-O(2)	108.8(2)	111.6(2)	106.9(4)
N(1)-P(1)-O(3)	108.4(2)	108.1(2)	106.9(3)
C(7)-O(2)-P(1)	119.3(3)	121.6(4)	131.5(9)
C(8)-O(3)-P(1)	124.4(4)	117.9(3)	118.1(6)
C(1)-N(1)-P(1)	128.6(3)	127.7(3)	129.2(6)
C(2)-C(1)-N(1)	119.9(4)	120.3(4)	119.2(8)
C(6)-C(1)-N(1)	120.5(4)	120.7(4)	119.8(9)
C(6)-C(1)-C(2)	119.6(4)	119.0(4)	120.9(8)
C(3)-C(2)-C(1)	118.2(4)	119.6(4)	118.1(9)
C(9)-C(2)-C(1)	123.4(4)	122.9(5)	123.3(8)
C(9)-C(2)-C(3)	118.4(4)	117.0(5)	118.5(10)
C(4)-C(3)-C(2)	121.6(5)	121.2(5)	121.3(10)
C(5)-C(4)-C(3)	120.0(5)	119.5(5)	119.6(9)
C(6)-C(5)-C(4)	119.9(5)	120.3(4)	121.0(9)
C(5)-C(6)-C(1)	120.6(5)	120.4(5)	119.0(9)
C(10)-C(9)-C(2)	111.5(5)	100.2(7)	111.0(9)

Table 7.12 Selected Torsion Angles (°) for both Structures with e.s.d's in Parentheses.

Angle	Structure (I)		Structure(II)
	Molecule A	Molecule B	
O(1)-P(1)-O(2)-C(7)	-62.6(5)	-57.1(5)	18.0(9)
O(3)-P(1)-O(2)-C(7)	175.0(5)	-178.6(5)	-105.8(9)
O(1)-P(1)-O(3)-C(8)	35.9(5)	57.1(5)	55.8(9)
O(2)-P(1)-O(3)-C(8)	158.8(5)	178.1(5)	180.0(9)
N(1)-C(1)-C(2)-C(3)	180.0(5)	180.0(5)	180.0(9)
N(1)-C(1)-C(2)-C(9)	0.0(5)	8.5(5)	-3.2(9)
C(6)-C(1)-C(2)-C(3)	0.0(5)	0.0(5)	1.3(9)
C(6)-C(1)-C(2)-C(9)	179.1(5)	-171.1(5)	177.3(9)
N(1)-C(1)-C(6)-C(5)	178.5(5)	180.0(5)	178.3(9)
C(2)-C(1)-C(6)-C(5)	-1.3(5)	0.0(5)	-2.3(9)
C(1)-C(2)-C(3)-C(4)	1.0(5)	0.0(5)	-0.9(9)
C(9)-C(2)-C(3)-C(4)	-177.9(5)	172.3(5)	-177.1(9)
C(1)-C(2)-C(9)-C(10)	-86.1(5)	-97.7(5)	-86.5(4)
C(3)-C(2)-C(9)-C(10)	92.7(5)	90.7(5)	89.5(9)
C(2)-C(3)-C(4)-C(5)	-1.3(5)	-0.9(5)	1.5(9)
C(3)-C(4)-C(5)-C(6)	0.0(5)	0.0(5)	-2.5(9)
C(4)-C(5)-C(6)-C(1)	1.0(5)	0.0(5)	2.9(9)
N(1)-P(1)-O(2)-C(7)	62.9(5)	69.4(5)	143.1(9)
N(1)-P(1)-O(3)-C(8)	-88.9(5)	-67.0(5)	-69.1(5)
O(1)-P(1)-N(1)-C(1)	-175.4(5)	-171.2(5)	-167.5(9)
O(2)-P(1)-N(1)-C(1)	55.2(5)	59.8(5)	65.3(9)
O(3)-P(1)-N(1)-C(1)	-48.8(5)	-44.1(5)	-40.8(9)
P(1)-N(1)-C(1)-C(2)	170.9(5)	152.8(5)	159.8(9)
P(1)-N(1)-C(1)-C(6)	-8.9(5)	-27.9(5)	-20.7(9)

The geometry of the phosphorus group is similar to that observed in the previous compounds. The average value of 1.461(5) Å for the P = O bond in the title compound is not significantly different (within experimental error) from the mean value of 1.456(4) Å found in the unsubstituted analogue. The angles between the groups attached to phosphorus atom conform to the pattern observed previously. The phosphorus atom is oriented away from the bulky *o*-ethyl substituent and so the latter group has little influence on the geometry of the phosphorus group. The P – O(2) – C(7) angle in structure (II) is 131.5(9)° and is much larger than the expected value of 120° for this angle. The O(1) – P(1) and O(2) – C(7) groups in this compound exhibit an eclipsed conformation (the O(1) – P(1) – O(2) – C(7) torsion angle is 18.0(9)°) leading to repulsions between O(1) and C(7). In comparison the P – O(2) – C(7) angle in structure (I) is approximately 120° with a O(1) -- P(1) – O(2) – C(7) torsion angle of approximately 58° (staggered). Both the P – N bond lengths (mean = 1.627(6) Å) and the N – C(1) bond lengths (mean = 1.424(5) Å) are not greatly different from the values observed in the previous structures. The C(1) – N(1) -- P bond angles (mean = 128.5(6)°) are also characteristically larger than the expected value of 120°. In structure (I) the phosphoramidate group is planar, however in structure (II) the phosphoramidate group is non-planar (the O(1) -- P(1) – N(1) – C(1) torsion angle is -167.5(9)°). In addition in structure (II) the P – N(1) -- C(1) bond angle is slightly larger than those in structure (II). The small differences in the geometry of the amide group in the two structures are possibly the result of the different constraints imposed by the different hydrogen bonding schemes in the two compounds. In carboxylic amides significant distortions of the amide group occur

in favour of hydrogen bonding (for example to achieve a linear hydrogen bond).[33]

The Benzene Ring Geometry

The effect of the σ-ethyl group on the ring geometry is expected to be small and this should be observed in terms of the internal angles of the ring.[69] The *ipso* angles of the amide groups, C(6) – C(1) -- C(2) (α_{NH}) are close to the standard value of 119.1(2)° for aniline derivatives.[50] The amide groups are coplanar with the ring, indicative of π interaction between the amide groups and the rings. The *ipso* angles of the ethyl substituents (C(1) – C(2) – C(3)) are smaller than 120°. In both structures the ethyl groups are bent away from C(1) towards C(3). The distortion of the angles defining the ethyl substituent are the result of repulsions in the crowded dimer. The deviations of the atoms from the least-squares planes calculated through the rings are given in Table 7.13. These deviations are small and the benzene rings essentially retain their ideal symmetry.

Hydrogen Bonding and Crystal Packing

Details of hydrogen bonding schemes are given in Table 7.14. Perspective views of the hydrogen bonding are given in Figures 7.3 and 7.4 respectively.

The N ---- O hydrogen bond distances in both compounds are virtually

Table 7.13 The Least-Squares Planes Calculated through the Benzene Rings and the Deviations of Selected Atoms from the Planes with e.s.d.'s in Parentheses.

Equations of the planes:

Plane 1: Structure (I),molecule A

$-3.725x + 0.609y + 23.840z = 1.076$

Plane 2: Structure (I),molecule B

$-7.674x + 5.976y + 6.309z = -3.467$

Plane 3: Structure (II)

$-4.85x + 7.36y + 6.81z = 0.20$

Deviations (Å):

	Plane 1	Plane 2	Plane 3
N(1)	-0.027(8)	0.000(8)	0.02(1)
*C(1)	-0.006(8)	-0.004(8)	0.01(1)
*C(2)	-0.002(8)	0.000(8)	0.00(1)
*C(3)	0.008(8)	0.004(8)	0.00(1)
*C(4)	-0.005(8)	-0.005(8)	-0.01(1)
*C(5)	-0.003(8)	0.002(8)	0.01(1)
*C(6)	0.008(8)	0.003(8)	-0.01(1)
C(9)	-0.035(8)	0.203(8)	0.08(1)
C(10)	-1.441(8)	-1.079(8)	1.44(1)

*Atoms used to calculate the least-squares plane

Table 7.14 Hydrogen Bonding Data and Close Contacts (Å) for both Structures with e.s.d.'s in Parentheses.

Hydrogen Bonding

	Structure (I)	Structure (II)
N(1A)-H(1A)	0.99(1) Å	1.00(1) A
N(1B)-H(1B)	0.99(1) Å	
O(1A).....N(1B)	2.889(4) Å	2.890(6) Å [1]
O(1B).....N(1B)	2.915(4) Å	
H(1A).....O(1B)	1.95(1) Å	1.94(1) Å [1]
H(1A).....O(1B)	1.95(1) Å	
O(1A)-H(1B)-N(1B)	156.6(9) °	159.8(9) °
O(1B)-H(1A)-N(1A)	164.3(9) °	

Close contacts

Structure (I)		Structure (II)	
O(3B)...C(7A)	3.412(6) [2]	O(1)...O(1)	3.25(1) [1]
O(1B)...O(1A)	3.235(6) [3]	O(1)...C(9)	3.37(1) [1]
O(1B)...C(9A)	3.284(6) [3]		

Symmetry code:

[1] -x -y -z

[2] 1-x -.5+y .5-z

[3] x y z

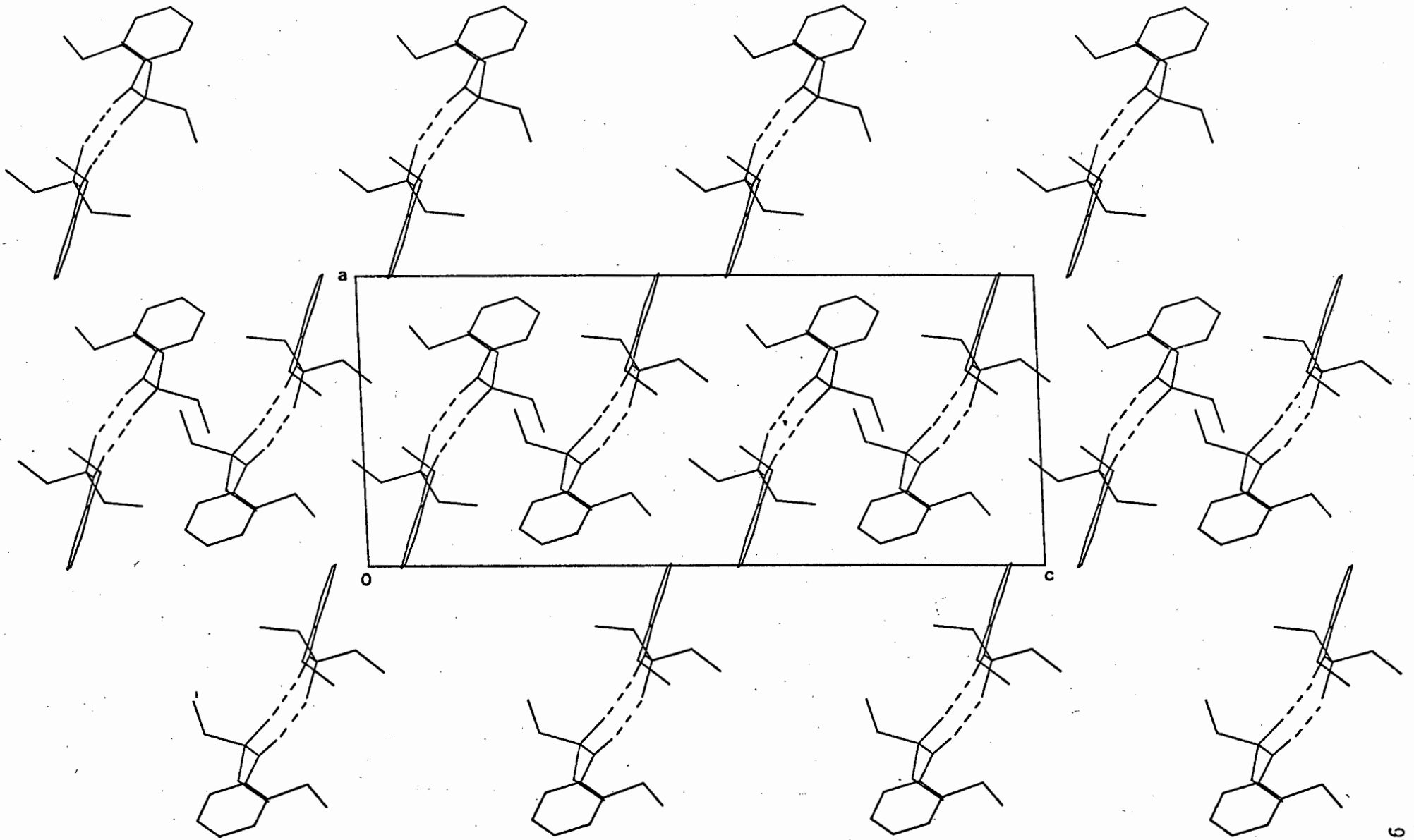


Figure 7.3 A View of the Crystal Packing in Structure (I)

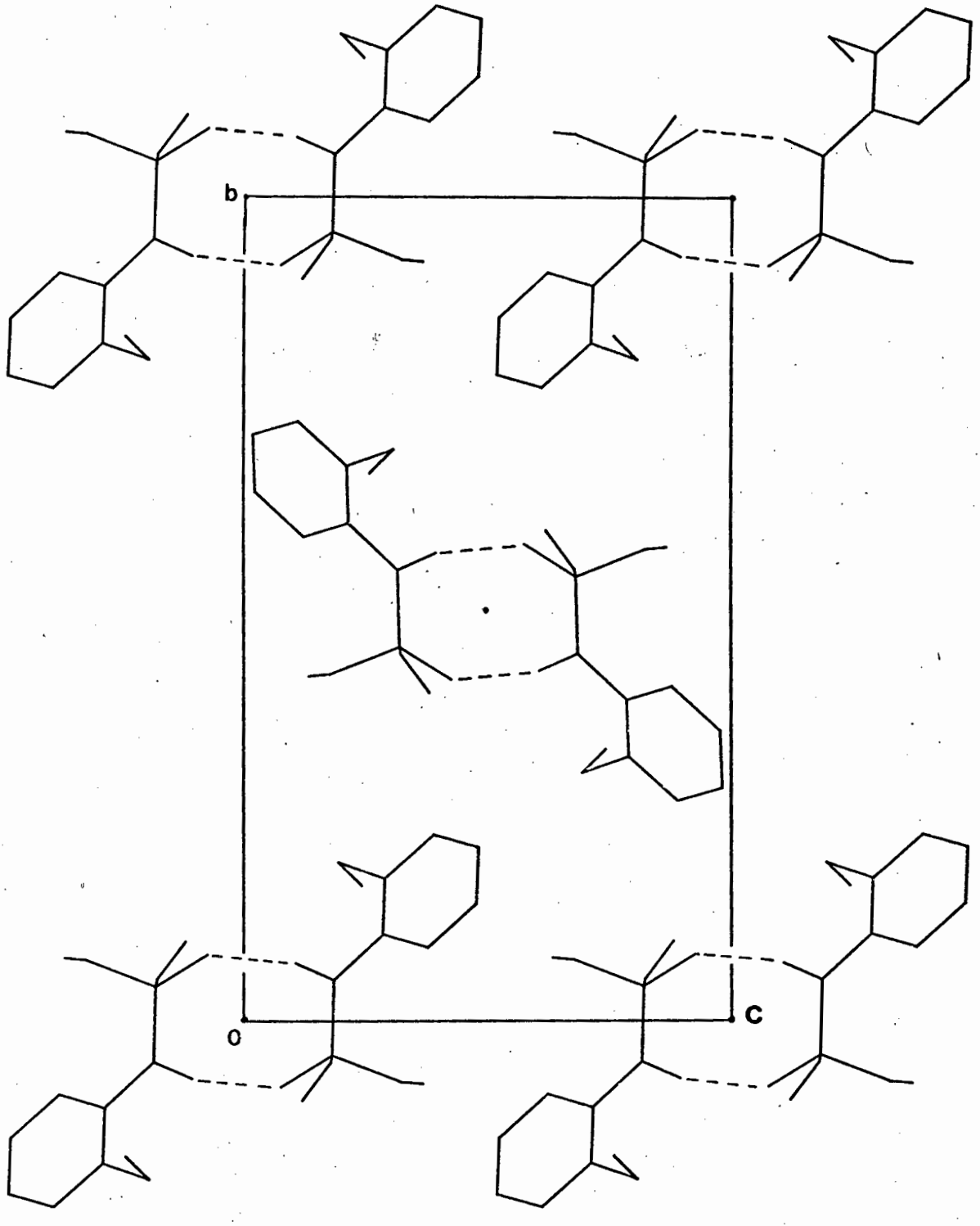


Figure 7.4 Crystal packing in structure (II)

identical and the mean value of 2.898(6) is within the range of values observed previously. In both structures the hydrogen bond angle (O --- H — N) is non-linear. The hydrogen bonding in structure (I), where two independent molecules dimerize is similar to that observed in the *p*-methoxy analogue. However, in the case of the latter compound the two hydrogen bonds are closer to 180° than in structure (I). Structure (II) may be compared with the unsubstituted analogue where a linear hydrogen bond is achieved. The presence of the *o*-ethyl substituent in the title compound results in dimers which are crowded and so steric factors prevent the achievement of a linear hydrogen bond. In the title compounds the hydrogen bonding situation that occurs in structure (I) appears to be more favourable since hydrogen bonding geometry is achieved with little distortion of the phosphoramidate geometry. In the centrosymmetric dimer (structure (II)) the constraints of symmetry require some distortion (from planarity) of the phosphoramidate group to achieve the observed hydrogen bond geometry.

Close contacts (<3.5 Å) are listed in Table 7.14.

7.4 Postscript

The question remains: why does the title compound result in two different crystal structures? The structures of the molecules are virtually identical and there are only small differences in the intermolecular interactions in the two cases. The two structures were obtained from different solvent systems; structure (I) from an oil

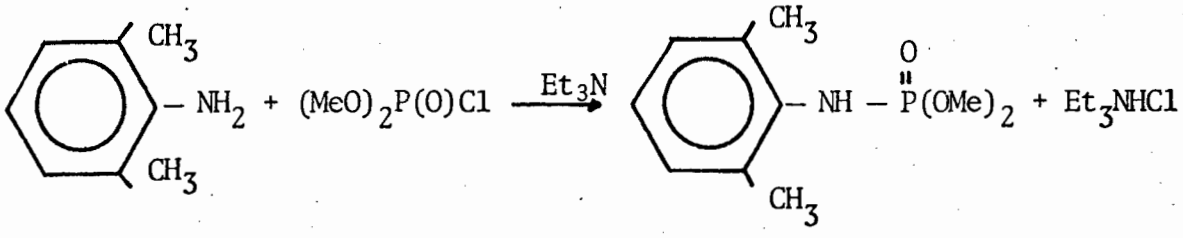
(from benzene) and structure (II) from a petroleum ether solution. The conditions prevailing in the two solutions just prior to crystallization are expected to be greatly different, resulting in two different structures. The way in which a mixture of compounds of different symmetries crystallize has been considered by a number of researchers particularly since a number of cases of the respontaneous resolution of racemic mixtures on crystallization have been reported. Unfortunately these phenomena are not yet fully understood.[163-165]

CHAPTER EIGHT

8. THE CRYSTAL AND MOLECULAR STRUCTURE OF DIMETHYL-N-(2,6-DIMETHYL-PHENYL)-PHOSPHORAMIDATE

8.1 Experimental

The title compound was synthesized according to the reaction:



The experimental procedure has been discussed previously in Chapter 4 in connection with the *p*-methoxy analogue.[38] The product was recrystallized from petroleum ether (60 - 90°C) as colourless needles. Found for $C_{10}H_{16}NO_3P$: C = 52.52%, H = 7.21%, N = 6.22%. Calculated C = 52.40 %, H = 7.04 %, N = 6.11 %.

The space group was assigned from preliminary oscillation and Weissenberg photography (*h*0*l*, *h*1*l*, *hk*0, *hk*1). The choice of $P2_1/c$[11] was confirmed during diffractometer data collection. The crystal data are listed in Table 8.1. Once again the absorption correction A* was equal to 1.0 over the entire range of θ scanned. The density was determined in a density column[39].

8.2 Solution and Refinement of the Structure

The structure was solved by the automatic centrosymmetric direct methods routine of the SHELX[12] program system, in which an *E*-map revealed the positions of most of the heavy atoms. The positions of the remaining heavy atoms as well as those of most of the hydrogen atoms, including

Table 8.1 Crystal data and Experimental and Refinement Parameters for the Structure Analysis

Crystal Data	
Molecular formula	$C_{10}H_{16}NO_3P$
M_r	229.2
Space group	$P2_1/c$
a	8.453(4) Å
b	14.231(7) Å
c	10.283(5) Å
β	108.95(2)°
V	1169.9 Å^3
D_m	1.28 Mgm^{-3}
D_c	1.30 Mgm^{-3} for $Z = 4$
$\mu(MoK_\alpha)$	0.178 mm^{-1}
μR_{min}, μR_{max}	0.01, 0.04
A*	1.0 %
F(000)	488
Data Collection	
Crystal dimensions	0.5 x 0.2 x 0.1 mm
Scan mode	ω - 2θ
Scan width	0.9° θ
Scan speed	0.03° θ s^{-1}
Range scanned (2θ)	6 - 46°
Stability of standard reflections	<1 %
Number of reflections collected	1600
Number of 'observed' reflections	1220 with $I_{rel} > 2\sigma I_{rel}$
Final Refinement	
Number of variables	154
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.061
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.065
Weighting scheme w	$(\sigma^2 F + .001F^2)^{-1}$
U_{iso}(aromatic H)	0.065(9) Å^2
U_{iso}(methyl H)	0.13(1) Å^2
U_{iso}(amide H)	0.07(2) Å^2

the amide hydrogen atom, were located in a difference electron-density map calculated after a number of cycles of least-squares refinement. In the final refinements the heavy atoms were treated anisotropically. The methyl hydrogen atoms were refined as rigid groups and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atom was constrained at 1.00 ± 0.01 Å from the nitrogen atom. The isotropic temperature factors of the methyl, aromatic and amide hydrogen atoms were treated as three separate parameters and refined independently.

In the final cycle of least-squares refinement the average shift to error ratio was less than 1.5 %. A final difference electron-density map calculated after the final cycle of least-squares refinement revealed no peaks of height > 0.9 e $Å^{-3}$. An analysis of variance calculated after the final least-squares refinement is given in Table 8.2 Lists of observed and calculated structure factors will be found in Appendix 8, final atomic coordinates in Tables 8.3 and 8.4 and anisotropic temperature factors in Table 8.5.

8.3 Description of the Structure and Discussion

A perspective view of the title compound is given in Figure 8.1. Principal bond lengths, angles and torsion angles are given in Tables8.6, 8.7 and 8.8 respectively.

The title compound differs from all the other phosphoramidates in the series in that the phosphoramidate group exhibits the *trans* orientation

Table 8.2 Analysis of Variance[a]

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	179	160	146	154	137	138	159	147	1220
V	110	112	103	95	101	116	101	107	106

(b) As a function of sin (θ)

sin(θ)	0	.18	.22	.25	.28	.30	.32	.34	.36	.38	.40
N	140	115	115	137	118	120	125	146	128	76	
V	147	142	94	97	90	94	94	91	83	103	

(c) As a function of $\sqrt{F/F_{max}}$

$\sqrt{F/F_{max}}$	0	.22	.24	.26	.28	.31	.34	.38	.43	.49	1.00
N	179	104	117	91	135	108	127	135	106	118	
V	114	100	102	106	119	118	109	92	83	104	

(c) As a function of |Miller index|

$\|h\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	106	206	197	180	167	134	110	72	41	7	0	0	0	0	0
V	108	104	109	116	100	93	89	109	119	174	0	0	0	0	0

$\|k\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	65	115	110	110	123	112	97	93	92	84	65	55	43	30	26
V	129	114	120	102	115	116	127	89	94	95	71	88	80	67	81

$\|l\|$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	89	153	174	149	149	124	108	92	84	61	35	2	0	0	0
V	111	133	104	107	116	98	103	80	93	78	80	4	0	0	0

[a] N = Number of reflections in the group; $V = 100[M(\Sigma \underline{w}|\underline{F}_o - \underline{F}_c|^2)/N\Sigma \underline{w}]$; M = Total number of reflections.

Table 8.3 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	x	y	z
P(1)	806(2)	2922(1)	1199(1)
O(1)	754(5)	2743(2)	2570(3)
O(2)	-667(5)	3462(3)	161(4)
O(3)	2262(4)	3669(3)	1317(3)
N(1)	1048(4)	1978(3)	404(3)
C(1)	1925(6)	1152(3)	1062(4)
C(2)	3561(6)	1002(3)	1087(5)
C(3)	4368(6)	184(4)	1654(5)
C(4)	3568(7)	-477(4)	2192(5)
C(5)	1949(7)	-329(4)	2181(5)
C(6)	1093(6)	492(3)	1609(4)
C(7)	-2304(8)	3005(5)	-424(9)
C(8)	2380(8)	4113(4)	102(6)
C(9)	4449(7)	1715(4)	478(7)
C(10)	-682(6)	624(4)	1597(5)

Table 8.4 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms.

	x	y	z
H(1)	90	216	-56
H(31)	564	6	168
H(41)	421	-112	262
H(51)	134	-85	262
H(71)	-335	348	-70
H(72)	-206	277	62
H(73)	-259	241	-111
H(81)	362	439	25
H(82)	149	468	-12
H(83)	205	363	-75
H(91)	357	224	-10
H(92)	508	140	-17
H(93)	536	205	135
H(101)	-148	73	55
H(102)	-76	123	221
H(103)	-108	1	201

Table 8.5 Anisotropic Temperature Factors ($Å^2 x 10^3$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
P(1)	64(1)	39(1)	27(1)	0(1)	17(1)	9(1)
O(1)	90(3)	47(2)	33(2)	6(2)	30(2)	-3(2)
O(2)	73(3)	50(2)	51(2)	2(2)	17(2)	14(2)
O(3)	65(2)	50(2)	45(2)	-3(2)	8(2)	-14(2)
N(1)	44(2)	32(2)	24(2)	0(2)	14(2)	0(2)
C(1)	39(3)	32(3)	25(2)	-4(2)	9(2)	0(2)
C(2)	37(3)	44(3)	39(3)	-1(2)	14(2)	-2(2)
C(3)	33(3)	51(3)	53(3)	1(3)	11(3)	8(3)
C(4)	49(4)	47(3)	51(3)	5(3)	7(3)	9(3)
C(5)	62(4)	42(3)	32(3)	2(2)	12(3)	0(3)
C(6)	37(3)	40(3)	28(3)	-4(2)	13(2)	-1(2)
C(7)	47(4)	69(4)	130(7)	2(4)	19(4)	-5(3)
C(8)	62(4)	69(4)	56(4)	19(3)	11(3)	-8(3)
C(9)	45(3)	58(4)	86(5)	4(3)	35(3)	-5(3)
C(10)	41(3)	53(3)	44(3)	-3(3)	18(3)	-8(3)

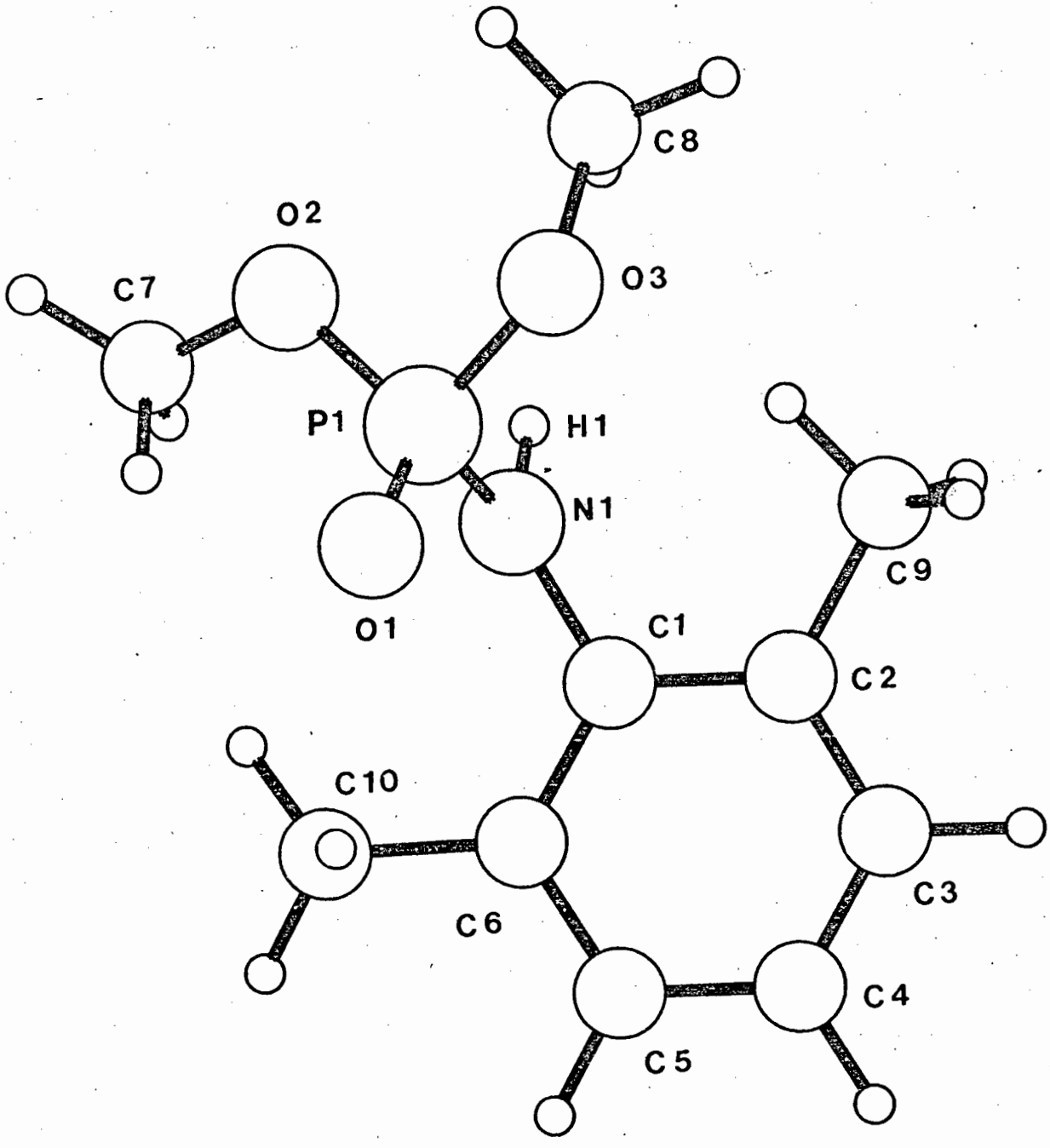


Figure 8.1 A perspective view of the title compound

Table 8.6 Principal Bond Lengths (Å) with e.s.d.'s in Parentheses.

P(1)-O(1)	1.448(4)
P(1)-O(2)	1.554(4)
P(1)-O(3)	1.600(4)
P(1)-N(1)	1.620(4)
C(7)-O(2)	1.469(7)
C(8)-O(3)	1.433(6)
C(1)-N(1)	1.436(5)
C(2)-C(1)	1.391(6)
C(6)-C(1)	1.397(6)
C(3)-C(2)	1.380(7)
C(9)-C(2)	1.513(6)
C(4)-C(3)	1.375(7)
C(5)-C(4)	1.380(7)
C(6)-C(5)	1.399(6)
C(10)-C(6)	1.508(6)

Table 8.7 Principal Bond Angles (°) with e.s.d's in in Parentheses.

O(1)-P(1)-O(2)	119.0(2)
O(1)-P(1)-O(3)	108.0(2)
O(1)-P(1)-N(1)	113.2(2)
O(3)-P(1)-O(2)	98.6(2)
N(1)-P(1)-O(2)	105.6(2)
N(1)-P(1)-O(3)	111.7(2)
C(7)-O(2)-P(1)	120.0(4)
C(8)-O(3)-P(1)	119.8(3)
C(1)-N(1)-P(1)	124.9(3)
C(2)-C(1)-N(1)	119.4(4)
C(6)-C(1)-N(1)	119.3(4)
C(6)-C(1)-C(2)	121.2(4)
C(3)-C(2)-C(1)	119.2(4)
C(9)-C(2)-C(1)	120.9(4)
C(9)-C(2)-C(3)	119.8(4)
C(4)-C(3)-C(2)	120.5(4)
C(5)-C(4)-C(3)	120.6(5)
C(6)-C(5)-C(4)	120.4(5)
C(5)-C(6)-C(1)	118.1(4)
C(10)-C(6)-C(1)	122.9(4)
C(10)-C(6)-C(5)	119.0(4)

Table 8.8 Selected Torsion Angles (°) with e.s.d's in Parentheses.

O(1)-P(1)-O(2)-C(7)	-71.2(4)
O(3)-P(1)-O(2)-C(7)	172.6(4)
N(1)-P(1)-O(2)-C(7)	57.2(4)
O(1)-P(1)-O(3)-C(8)	-167.7(4)
O(2)-P(1)-O(3)-C(8)	-43.4(4)
N(1)-P(1)-O(3)-C(8)	67.2(4)
O(1)-P(1)-N(1)-C(1)	-29.9(4)
O(2)-P(1)-N(1)-C(1)	-161.7(4)
O(3)-P(1)-N(1)-C(1)	92.3(4)
P(1)-N(1)-C(1)-C(2)	-100.0(4)
P(1)-N(1)-C(1)-C(6)	82.9(4)
N(1)-C(1)-C(2)-C(3)	-176.6(4)
N(1)-C(1)-C(2)-C(9)	2.3(4)
C(6)-C(1)-C(2)-C(3)	0.0(4)
C(6)-C(1)-C(2)-C(9)	180.0(4)
N(1)-C(1)-C(6)-C(5)	176.8(4)
N(1)-C(1)-C(6)-C(10)	-2.7(4)
C(2)-C(1)-C(6)-C(5)	0.0(4)
C(2)-C(1)-C(6)-C(10)	180.0(4)
C(1)-C(2)-C(3)-C(4)	0.0(4)
C(9)-C(2)-C(3)-C(4)	-179.0(4)
C(2)-C(3)-C(4)-C(5)	0.0(4)
C(3)-C(4)-C(5)-C(6)	0.0(4)
C(4)-C(5)-C(6)-C(1)	0.0(4)
C(4)-C(5)-C(6)-C(10)	180.0(4)

of the P = O and N – H groups. Repulsion between the phosphate ester groups and the 2,6-dimethyl substituents prevents the formation of the *syn* orientation and hence prevents dimerization. The phosphate ester groups are twisted so as to achieve the greatest separation from the methyl groups on the benzene ring. The P = O group is *cis* to the benzene ring similar to the so called *endo* conformation found in carboxylic amides.[70] This situation prevents the phosphoramidate group from achieving coplanarity with the benzene ring. However, MO calculations at the CNDO/2 level of approximation on acetanilide have indicated that electrostatic attraction between the oxygen atom and the slightly electron deficient C(1) atom is an important source of stabilization energy for the *endo* conformation.[71]

The distortion of the tetrahedral angles about the phosphorus atom are particularly severe. The O(1) -- P – O(2) bond angle is 119.0(2)°, the largest yet found in this series. Once again the O(2) – P – O(3) angle is considerably compressed to 98.6(3)°. The P = O(1) bond length at 1.448(4) Å is somewhat shorter than the values (∿1.460 Å) found in the previous structures. The P – N and N – C(1) bond lengths are within the range of values observed in the previous structures in the series. The P – N – C(1) bond angle, 124.9(3)°, is smaller than the values found in the phosphoramidates discussed previously. The phosphoramidate group is twisted at 83° out of the plane of the benzene ring (the P(1) – N(1) – C(1) -- C(6) torsion angle is 82.9(4)°), thus reducing the repulsion between the phosphoryl group and the *ortho* substituents on the ring. The reduced steric interactions of these two groups results in a reduction in the size of the P – N – C(1) angle over that observed in the *cis* phosphoramidates. The twist of the phosphoramidate group with respect to the benzene ring effectively eliminates any

π-interaction between these groups.

The phosphoramidate group is distorted out of planarity (the O(1) – P(1) – N(1) – C(1) torsion angle is -29.9(4)°). In the *cis* phosphoramidates discussed previously this group was much less distorted from planarity. The distortion of the phosphoramidate group in this compound is possibly as a result of the crowding introduced by the *o*-methyl groups or as a result of intermolecular hydrogen bonding effects[33].

The Benzene Ring Geometry

It is well known that certain substituents like methyl and *tert*-butyl groups, although lacking unshared electron pairs, are able to cause a perturbation of the π-electron system of the ring very much like that caused by π donors. This has been attributed to hyper-conjugation, but MO calculations carried out on toluene support the idea that the π-electron system of the ring is polarised by the methyl group, with little net electron transfer[72]. It is therefore expected that angular distortions would occur in the benzene ring of the title compound, similar to those caused by π-donor functional groups. In the title compound such distortions are observed; the *ipso* angles (α_{CH_3}) of the two methyl groups (C(5) – C(6) – C(1) and C(1) – C(2) – C(3)) are 118.1(4)° and 119.2(4)° respectively. These values are slightly larger than the value of 117.5(3)° determined by low temperature X-ray crystallography for toluene[73]. In the title compound the α_{CH_3} angles are enlarged due to the influence of the opposite methyl group on these angles[69].

The *ipso* angle of the amide group, α_{NH} (C(6) – C(1) – C(2)), is larger than the average value of 119.1(2)° found for analine derivatives[50]. Since the phosphoramidate group and the ring are almost at 90° to each other the value of α_{NH} for this compound of 121.2(4)° is free of π-acceptor or donor affects. This angle is however enlarged by the presence of the methyl substituents; in toluene the angles adjacent to α_{CH_3} are 121.1(4)° and 121.3(4)°.[73]

The substituents do not have a very great affect on the planarity of the ring and Table 8.9 shows that the ring is essentially planar. Once again it is evident bond angles are more sensitive to substituent affects than inter-atomic distances. Both methyl substituents are bent away from C(1) below the ring.

Hydrogen Bonding and Crystal Packing

A perspective view of the hydrgen bonding scheme in this compound is given in Figure 8.2. The hydrogen bonding details are listed in Table 8.10.

The *trans* orientation of the phosphoramidate group in the title compound permits the formation of a polymeric hydrogen bonding motif. There is an intermolecular network of N – H --- O = P – hydrogen bonds parallel to the *c* axis and related by the *c* glide plane. The glide motif is common in carboxylic amides[62]. The hydrogen bond distance, N ---- O, is similar to that observed in the previous structures. The N – H ---- O hydrogen bond is bent. Carboxylic amides are generally planar and interlayer interactions have been shown to be important

Table 8.9 The Least-Squares Plane Calculated through the Benzene Ring and the Deviations of Selected Atoms from the Plane with e.s.d.'s in Parentheses.

Equation of the plane:

$0.486x + 6.379y + 8.484z = 1.732$

Deviations (Å):

N(1)	-0.077(6)
*C(1)	-0.003(6)
*C(2)	0.002(6)
*C(3)	0.001(6)
*C(4)	-0.003(6)
*C(5)	0.003(6)
*C(6)	0.000(6)
C(9)	-0.016(6)
C(10)	-0.012(6)

*Atoms used to calculate the least-squares plane

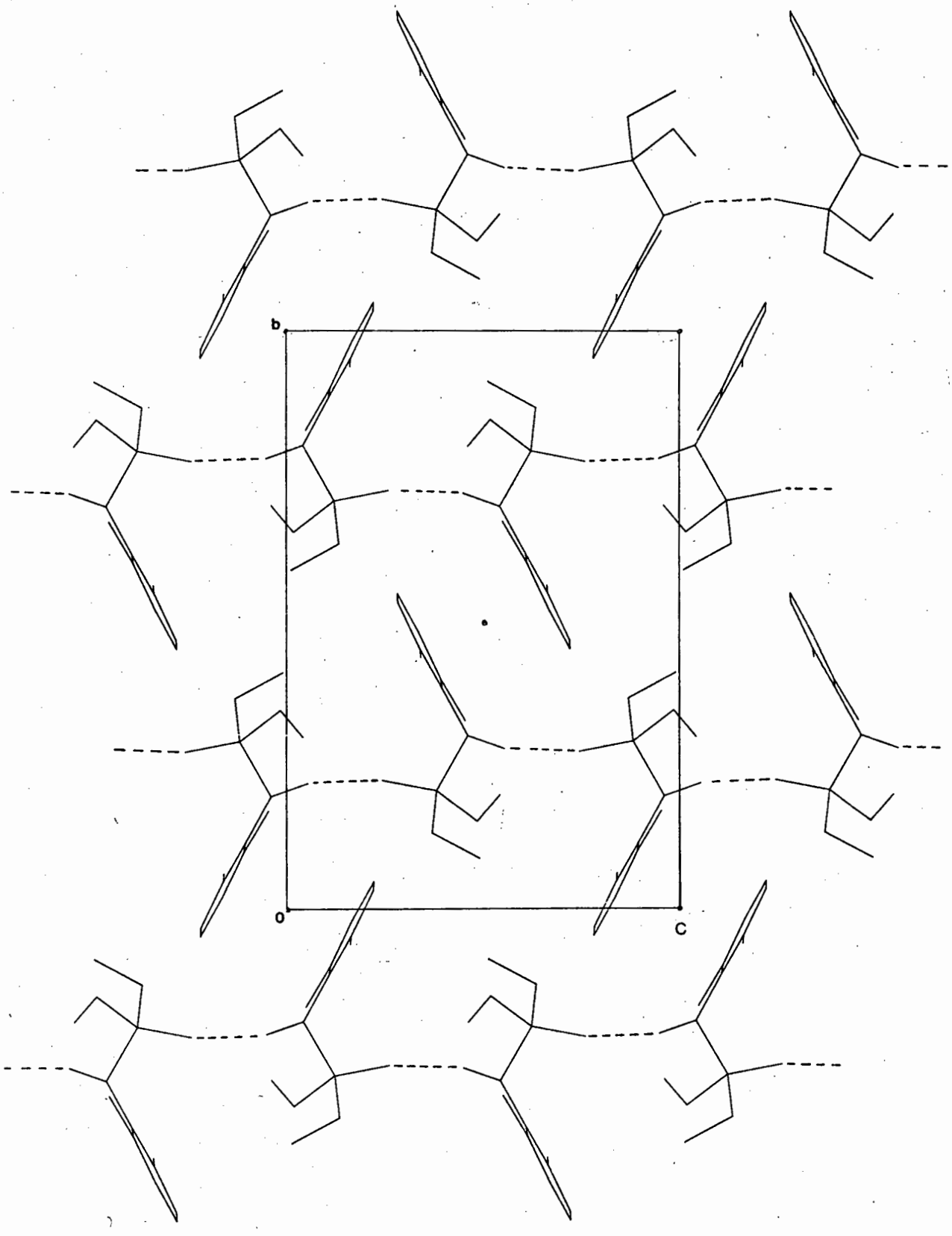


Figure 8.2 A projection of the crystal structure showing the hydrogen bonding

Table 8.10 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen bonding	
N(1)-H(1)	0.9(9) Å
N(1).....O(1) [1]	2.87(1) Å
H(1).....O(1) [1]	1.89(1) Å
N(1)-H(1)-O(1)	168.5(9)°
Close contacts	
C(8).....C(6) [2]	3.444(5)
C(8).....C(5) [2]	3.382(5)

Symmetry code:

[1] x .5-y -.5+z

[2] -x 1-y -z

contributors to the overall crystal packing and hydrogen bonding motif[65]. Since the title compound is non-planar there is only limited layer overlap. This overlap is limited to overlap between the benzene rings across centres of symmetry. The close contacts (<3.5 Å) are listed in Table 8.10.

CHAPTER NINE

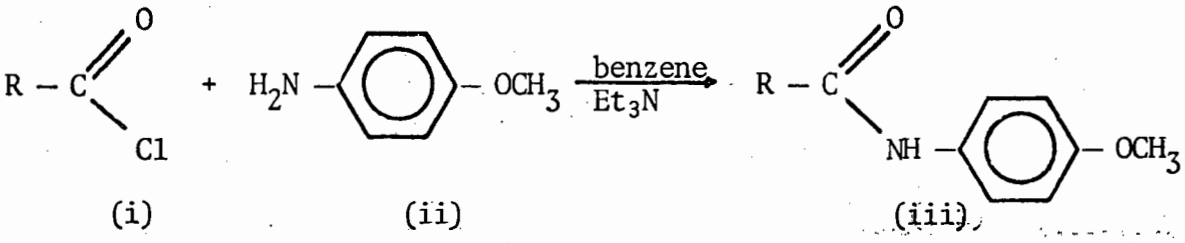
9. THE CRYSTAL AND MOLECULAR STRUCTURE OF *p*-METHOXYACETANILIDE (I) AND BENZ-*p*-METHOXYANILIDE (II)

9.1 Introduction

The crystal structure of compound (I) was published [74] shortly after it was solved in this laboratory. The published structure was solved from photographic data and so it was decided to complete the second refinement using diffractometer data. Compound (II) was solved to compare structure features with benz-*p*-nitroanilide which will be described in the next chapter.

9.2 Experimental

The title compounds were synthesized according to the reaction below:



(i) (ii) (iii) (iv)

(I) R = CH_3

(II) R = Ph

Equimolar amounts of the acid chloride (i), the substituted aniline (ii) and triethylamine were dissolved in benzene and stirred. The resulting precipitate of the triethylamine hydrochloride (iv) was separated from the benzene solution. The required product (iii) was obtained from the benzene solution after the solvent had been removed under reduced

pressure. Suitable crystals (colourless needles) were obtained after slow recrystallization from ethanol. Found for compound (I) : $C_9H_{11}NO_2$; C = 65.51%, H = 6.92%, N = 8.63%. Calculated: C = 65.45%, H = 6.67%, N = 8.48%. Found for compound (II): $C_{14}H_{13}NO_2$; C = 74.06%, H = 6.02%, N = 6.31%. Calculated: C = 74.01%, H = 5.73%, N = 6.17%. The space group of compound (I) was assigned from preliminary oscillation, Weissenberg ($0kl$, $1kl$) and precession ($h0l$, $h1l$, $hk0$) photography. The space group of compound (II) was assigned from preliminary oscillation and Weissenberg ($0kl$, $1kl$, $h0l$, $h1l$) photography. In both cases the 1 large cell parameter precluded the measurement of background radiation for individual reflections. A background vs.θ curve (obtained from unobserved reflections) was thus used to estimate the background for all reflections. The crystal data are listed in Table 9.1. The crystals of compound (II) were rather poor and coupled with the method of background radiation measurement resulted in an inferior data set for compound (II) in comparison with compound (I). It was not necessary to apply an absorption correction to the data because the absorption correction factor A* remained equal to 1.0 over the range of θ scanned. The density was determined in a density column.[39]

9.3 Solution and Refinement of the Structures

Both structures were solved by the automatic centrosymmetric direct methods routine of the SHELX[12] program system, in which *E*-maps revealed the positions of all the heavy atoms. The majority of the hydrogen atoms, including the amide hydrogen atoms, were located in difference electron-density maps calculated after a number of cycles of least-squares

Table 9.1 Crystal Data and Experimental and Refinement Parameters for the Structure Analysis

	Compound (I)	Compound (II)
Crystal Data		
Molecular formula	$C_9H_{11}NO_2$	$C_{14}H_{13}NO_2$
M_r	165.2	227.3
Space group	*Pbca*	$P2_1/c$
a (Å)	7.623(4)	26.74(1)
b (Å)	24.89(1)	5.252(3)
c (Å)	9.227(4)	8.145(4)
β (°)	-	97.23(2)
V (Å^3)	1752.8	1135.1
D_m (Mgm^{-3})	1.23	1.31
D_c (Mgm^{-3})	1.26 (Z = 8)	1.33 (Z = 4)
$\mu(MoK_\alpha)$ (mm^{-1})		
μR_{min}, μR_{max}	0.005, 0.013	0.003, 0.010
A*	1.0	1.0
F(000)	704	704
Data Collection		
Crystal dimensions (mm)	0.2 x 0.3 x 0.5	0.4 x 0.4 x 0.1
Scan mode	ω - 2θ	ω - 2θ
Scan width (°θ)	0.4	1.4
Scan speed (°θs^{-1})	0.013	0.05
Range scanned (2θ) (°)	6 - 46	6 - 46
Stability of standard reflections (%)	<1	<1.5
Number of reflections collected	1212	1492
Number of 'observed' reflections with $I_{rel} > 2\sigma I_{rel}$	1035	1202
Final refinement		
Number of variables	121	163
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.047	0.106
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.043	0.107
Weighting scheme w	$(\sigma^2 F)^{-1}$	$(\sigma^2 F + 0.002F^2)^{-1}$
U_{iso}(aromatic H) (Å^2)	0.07(1)	0.06(1)
U_{iso}(methyl H) (Å^2)	0.11(1)	0.03(1)
U_{iso}(amide H) (Å^2)	0.09(1)	0.11(2)

refinement. The final refinements were carried out with all the heavy atoms treated anisotropically, the methyl groups refined as rigid groups and the aromatic hydrogen atoms constrained at 1.08 Å from their respective carbon atoms, their positions dictated by the geometry of the molecules. The amide hydrogen atoms were constrained at 1.00 ± 0.01 Å from their nitrogen atoms. The isotropic temperature factors of the methyl, aromatic and amide hydrogen atoms were treated as three separate parameters and refined independently.

In the final cycle of least-squares refinement the average shift to error ratio was less than 1 % in both compounds. Difference electron density maps calculated after the final least-squares refinements revealed no peaks >0.1 e $Å^{-3}$ in both structures. Details of the final refinements are given in Table 9.1, analyses of variance calculated after the final refinements in Tables 9.2 and 9.3 and lists of observed and calculated structure factors in Appendix 9. Fractional atomic coordinates and temperature factors are given in Tables 9.4 to 9.9 inclusive.

9.4 Description of the Structures and Discussion

Perspective views of both compounds are given in Figures 9.1 and 9.2. Principal bond lengths, angles and torsion angles are given in Tables 9.10, 9.11 and 9.12 respectively.

Both compounds crystallize in the *endo* conformation in which the C = O groups are *cis* to the benzene ring and the bulky groups attached to C(1)

Table 9.2 Analysis of Variance[a] for Compound (I)

(a) By parity groups

	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	190	108	125	107	147	120	111	127	1035
V	69	75	67	67	59	65	65	64	67

(b) As a function of sin (θ)

sin(θ)	0	.18	.22	.26	.28	.31	.34	.36	.38	.40
N	111	98	125	83	147	58	111	106	127	69
V	116	79	65	59	63	63	58	47	37	33

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.16	.19	.21	.24	.26	.29	.33	.38	.47	1.00
N	119	120	77	123	81	118	104	99	95	99	
V	60	52	55	57	59	65	64	64	65	109	

(d) As a function of |Miller Index|

$\lvert h \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	106	172	196	143	159	101	98	46	14	0	0	0	0	0	0
V	67	83	77	63	61	55	47	38	41	0	0	0	0	0	0

$\lvert k \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	33	54	61	50	58	52	60	51	57	50	55	40	48	36	330
V	108	107	82	59	69	65	66	57	59	56	64	74	55	48	57

$\lvert l \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	85	154	162	139	131	114	93	71	53	27	6	0	0	0	0
V	97	65	74	70	58	65	55	48	53	42	36	0	0	0	0

[a] N = Number of reflections in the group; $V = 100[M\Sigma(w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 9.3 Analysis of Variance[a] for Compound (II)

(a) By parity groups

	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	194	190	155	150	103	107	151	152	1202
V	123	125	117	117	118	131	135	130	125

(b) As a function of sin (θ)

sin(θ)	0	.18	.22	.25	.28	.30	.32	.34	.36	.38	.40
N	140	115	116	142	102	118	124	138	140	67	
V	177	135	125	114	123	121	119	102	97	104	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.21	.24	.26	.28	.31	.34	.38	.43	.52	1.00
N	132	169	104	90	129	122	104	120	115	117	
V	102	121	140	116	129	97	123	117	128	164	

(d) As a function of |Miller Index|

$\lvert h \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	27	56	60	56	62	63	60	57	62	57	61	64	50	46	421
V	127	131	113	172	146	151	125	110	138	98	113	97	108	100	122

$\lvert k \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	174	312	270	231	150	65	0	0	0	0	0	0	0	0	0
V	126	130	136	123	96	102	0	0	0	0	0	0	0	0	0

$\lvert l \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	124	185	237	164	186	108	107	56	35	0	0	0	0	0	0
V	130	129	131	149	119	102	98	112	90	0	0	0	0	0	0

[a] N = Number of reflections in the group; $V = 100[M\Sigma(\underline{w}|\underline{F}_o-\underline{F}_c|^2)/N\Sigma\underline{w}]$; M = Total number of reflections.

Table 9.4 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms of Compound (I) with e.s.d.'s in Parentheses.

	x	y	z
O(1)	1468(2)	-83(1)	2574(2)
O(2)	286(2)	2435(1)	1402(2)
N(1)	2084(3)	295(1)	394(2)
C(1)	2071(3)	-119(1)	1343(3)
C(2)	1340(4)	2848(1)	784(4)
C(11)	1556(3)	833(1)	659(2)
C(12)	2215(3)	1227(1)	-264(2)
C(13)	1812(3)	1764(1)	-65(3)
C(14)	724(3)	1911(1)	1074(3)
C(15)	3(3)	1521(1)	1954(3)
C(16)	416(3)	983(1)	1754(2)
C(21)	2866(3)	-635(1)	799(3)

Table 9.5 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms of Compound (II) with e.s.d.'s in Parentheses.

	x	y	z
O(1)	2845(1)	8672(8)	4257(6)
O(2)	614(1)	5274(9)	1302(5)
N(1)	2622(2)	4475(10)	4170(6)
C(1)	2951(2)	6439(11)	4524(7)
C(2)	323(2)	7418(14)	1658(9)
C(11)	2105(2)	4785(11)	3461(6)
C(12)	1903(2)	2987(11)	2344(8)
C(13)	1397(2)	3208(11)	1628(7)
C(14)	1114(2)	5194(12)	2092(7)
C(15)	1308(2)	6971(11)	3229(7)
C(16)	1814(2)	6735(11)	3942(7)
C(21)	3483(2)	5648(10)	5232(7)
C(22)	3581(2)	3533(12)	6183(8)
C(23)	4084(2)	2941(13)	6834(8)
C(24)	4461(2)	4572(14)	6489(8)
C(25)	4367(2)	6670(13)	5527(9)
C(26)	3863(2)	7267(12)	4886(8)

Table 9.6 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms of Compound (I).

	x	y	z
H(1)	255	23	-60
H(21)	87	322	127
H(22)	119	287	-38
H(23)	271	279	105
H(121)	306	111	-115
H(131)	233	207	-79
H(151)	-89	163	281
H(161)	-15	68	245
H(211)	321	-63	-34
H(212)	192	-95	100
H(213)	403	-71	144

Table 9.7 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms of Compound (II).

	x	y	z
H(1)	274	268	421
H(21)	-3	699	93
H(22)	47	919	124
H(23)	27	759	294
H(121)	213	141	202
H(131)	124	183	73
H(151)	108	852	357
H(161)	197	809	486
H(221)	328	231	644
H(231)	417	126	758
H(241)	485	416	700
H(251)	467	788	525
H(261)	378	895	414

Table 9.8 Anisotropic Temperature Factors ($Å^2 x10^3$) of the Heavy Atoms of Compound (I) with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
O(1)	77(1)	67(1)	41(1)	10(1)	3(1)	10(1)
O(2)	68(1)	49(1)	90(1)	-1(1)	8(1)	9(1)
N(1)	54(1)	45(1)	37(1)	-1(1)	2(1)	2(1)
C(1)	46(2)	52(2)	41(1)	3(1)	-9(1)	-3(1)
C(2)	89(2)	50(2)	114(3)	7(2)	7(2)	2(2)
C(11)	40(1)	45(1)	38(1)	0(1)	-4(1)	1(1)
C(12)	51(2)	52(2)	41(1)	3(1)	5(1)	1(1)
C(13)	55(2)	52(2)	50(1)	9(1)	3(1)	1(1)
C(14)	47(1)	46(1)	56(2)	-1(1)	-5(1)	8(1)
C(15)	45(2)	58(2)	52(1)	0(1)	7(1)	5(1)
C(16)	45(1)	53(2)	45(1)	2(1)	6(1)	0(1)
C(21)	68(2)	48(1)	56(2)	-4(1)	-5(1)	6(1)

Table 9.9 Anisotropic Temperature Factors ($Å^2 x 10^3$) of the Heavy Atoms of Compound (II) with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
O(1)	35(2)	42(3)	109(4)	5(3)	-19(2)	3(2)
O(2)	27(2)	70(3)	64(3)	-8(2)	-14(2)	1(2)
N(1)	26(3)	37(3)	56(3)	-2(2)	-13(2)	-1(2)
C(1)	33(3)	34(4)	50(4)	0(3)	-10(3)	6(3)
C(2)	35(4)	68(5)	72(5)	-5(4)	-11(3)	11(3)
C(11)	27(3)	39(3)	36(3)	3(3)	-7(2)	-6(3)
C(12)	36(3)	45(4)	49(4)	-7(3)	-1(3)	0(3)
C(13)	33(3)	48(4)	42(3)	-9(3)	-7(3)	-5(3)
C(14)	27(3)	50(4)	41(3)	5(3)	-4(2)	-3(3)
C(15)	30(3)	49(4)	45(4)	1(3)	-2(3)	4(3)
C(16)	38(3)	45(4)	42(4)	-3(3)	-5(3)	1(3)
C(21)	27(3)	34(3)	47(3)	-6(3)	-7(2)	2(3)
C(22)	38(3)	53(4)	51(4)	-1(3)	-8(3)	4(3)
C(23)	52(4)	56(4)	55(4)	4(3)	-15(3)	13(3)
C(24)	28(3)	67(5)	63(4)	-9(4)	-15(3)	9(3)
C(25)	35(3)	62(4)	64(4)	-5(4)	-2(3)	1(3)
C(26)	34(3)	52(4)	53(4)	3(3)	-4(3)	1(3)

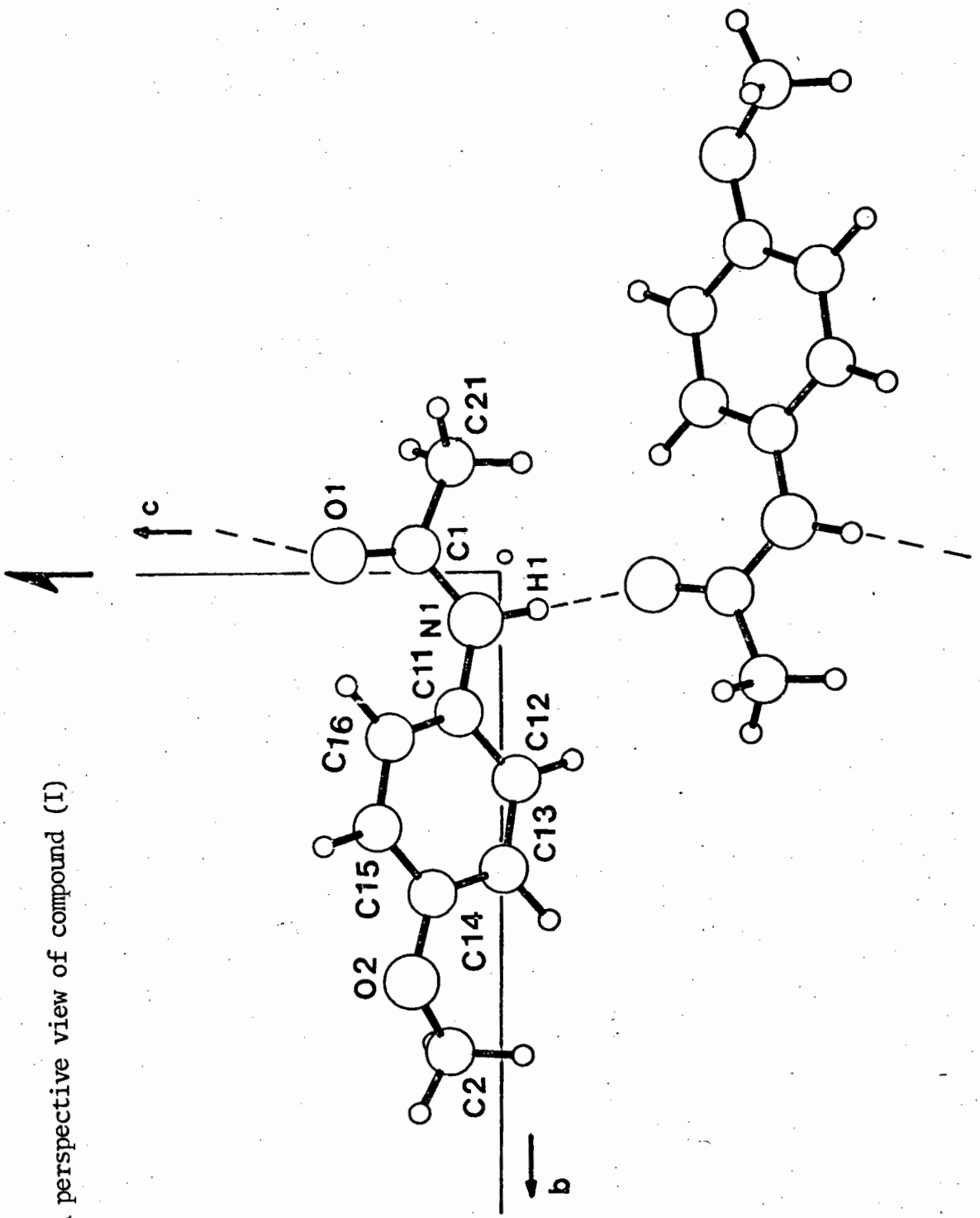


Figure 9.1 A perspective view of compound (I)

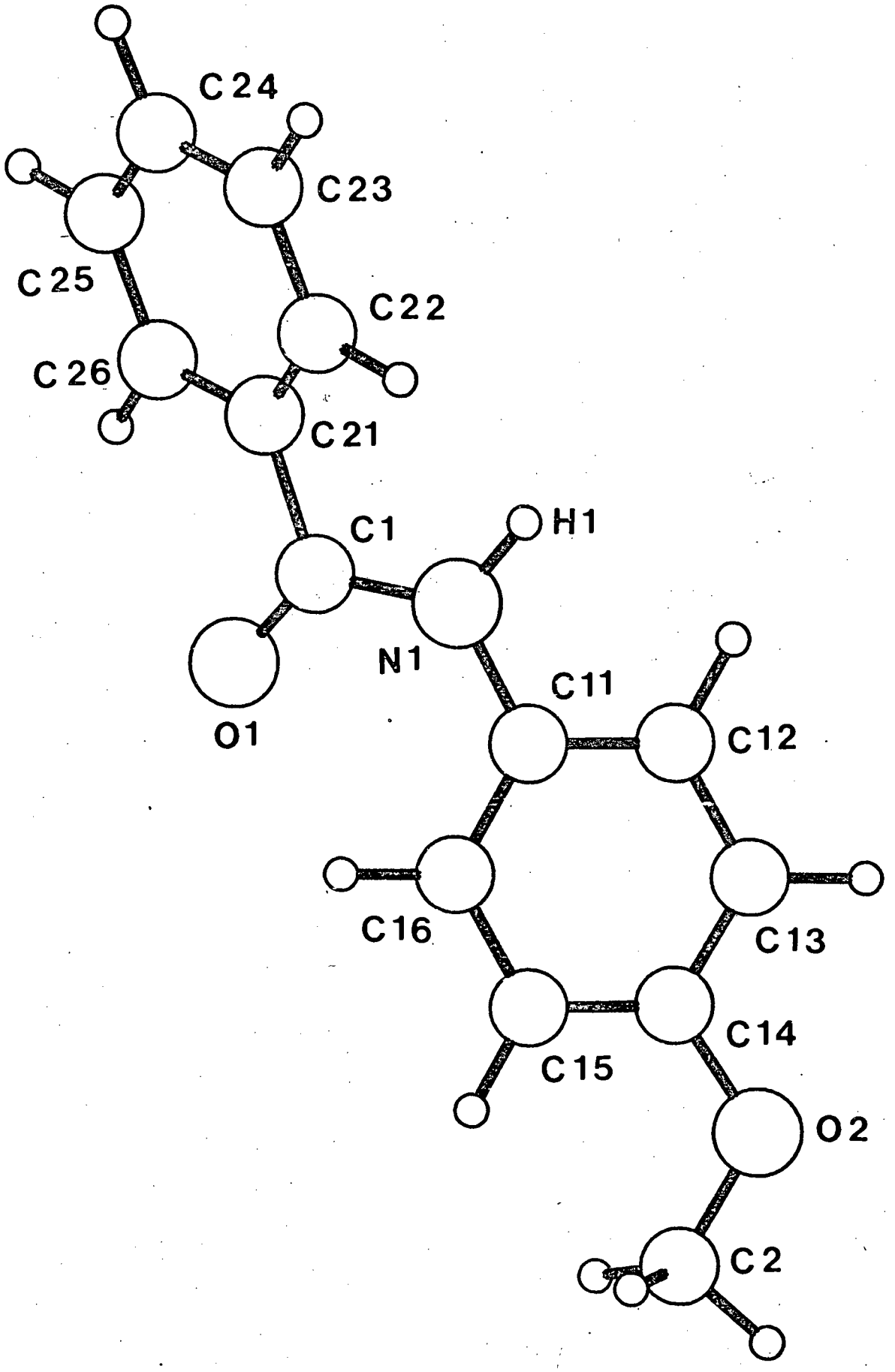


Figure 9.2 A perspective view of compound (II)

Table 9.10 Principal Bond Lengths (Å) with e.s.d. s in Parentheses.

Bond	Compound (I)	Compound (II)
C(1)-O(1)	1.229(3)	1.220(7)
C(2)-O(2)	1.424(3)	1.418(8)
C(14)-O(2)	1.379(3)	1.409(6)
C(1)-N(1)	1.352(3)	1.362(7)
C(11)-N(1)	1.421(3)	1.439(6)
C(21)-C(1)	1.505(3)	1.525(7)
C(12)-C(11)	1.392(3)	1.375(8)
C(16)-C(11)	1.385(3)	1.372(8)
C(13)-C(12)	1.385(3)	1.409(8)
C(14)-C(13)	1.388(3)	1.370(8)
C(15)-C(14)	1.381(3)	1.370(8)
C(16)-C(15)	1.387(3)	1.411(8)
C(22)-C(21)		1.361(8)
C(26)-C(21)		1.382(8)
C(23)-C(22)		1.415(8)
C(24)-C(23)		1.380(9)
C(25)-C(24)		1.357(9)
C(26)-C(25)		1.417(8)

Table 9.11 Principal Bond Angles (°) with e.s.d. s in Parentheses.

Angle	Compound (I)	Compound (II)
C(14)-O(2)-C(2)	117.2(2)	116.3(5)
C(11)-N(1)-C(1)	127.2(2)	124.1(5)
N(1)-C(1)-O(1)	123.0(2)	124.2(5)
C(21)-C(1)-O(1)	121.5(2)	120.9(5)
C(21)-C(1)-N(1)	115.5(2)	114.9(5)
C(12)-C(11)-N(1)	117.1(2)	117.8(5)
C(16)-C(11)-N(1)	123.9(2)	121.4(5)
C(16)-C(11)-C(12)	119.0(2)	120.7(5)
C(13)-C(12)-C(11)	121.2(2)	119.7(5)
C(14)-C(13)-C(12)	119.2(2)	119.0(5)
C(13)-C(14)-O(2)	124.0(2)	115.0(5)
C(15)-C(14)-O(2)	116.1(2)	123.0(5)
C(15)-C(14)-C(13)	119.9(2)	122.0(5)
C(16)-C(15)-C(14)	120.7(2)	118.7(5)
C(15)-C(16)-C(11)	120.0(2)	119.9(5)
C(22)-C(21)-C(1)		122.8(5)
C(23)-C(22)-C(21)		119.9(6)
C(24)-C(23)-C(22)		118.1(6)
C(25)-C(24)-C(23)		122.3(5)
C(26)-C(25)-C(24)		119.4(6)
C(25)-C(26)-C(21)		118.6(6)
C(26)-C(21)-C(1)		115.6(5)
C(26)-C(21)-C(22)		121·6(5)

Table 9.12 Selected Torsion Angles (°) with e.s.d.'s in Parentheses.

Torsion angle	Compound (I)	Compound (II)
C(2)-O(2)-C(14)-C(13)	-16.5(2)	-175.2(6)
C(2)-O(2)-C(14)-C(15)	163.9(2)	7.0(6)
C(11)-N(1)-C(1)-O(1)	5.0(2)	-1.1(6)
C(11)-N(1)-C(1)-C(21)	-174.5(2)	-178.6(6)
C(1)-N(1)-C(11)-C(12)	158.5(2)	143.5(6)
C(1)-N(1)-C(11)-C(16)	-22.7(2)	-39.8(6)
O(1)-C(1)-C(21)-C(22)		151.2(6)
O(1)-C(1)-C(21)-C(26)		-26.9(6)
N(1)-C(1)-C(21)-C(22)		-31.2(6)
N(1)-C(1)-C(21)-C(26)		150.7(6)
N(1)-C(11)-C(12)-C(13)	-178.2(2)	180.0(6)
C(16)-C(11)-C(12)-C(13)	2.9(2)	2.8(6)
N(1)-C(11)-C(16)-C(15)	178.6(2)	-177.1(6)
C(12)-C(11)-C(16)-C(15)	-2.6(2)	0.0(6)
C(11)-C(12)-C(13)-C(14)	0.0(2)	-1.4(6)
C(12)-C(13)-C(14)-O(2)	177.7(2)	180.0(6)
C(12)-C(13)-C(14)-C(15)	-2.7(2)	-2.6(6)
O(2)-C(14)-C(15)-C(16)	-177.4(2)	-177.0(6)
C(13)-C(14)-C(15)-C(16)	2.9(2)	5.3(6)
C(14)-C(15)-C(16)-C(11)	0.0(2)	-3.8(6)
C(1)-C(21)-C(22)-C(23)		-178.5(6)
C(26)-C(21)-C(22)-C(23)		0.0(6)
C(1)-C(21)-C(26)-C(25)		179.1(6)
C(21)-C(22)-C(23)-C(24)		0.0(6)
C(22)-C(23)-C(24)-C(25)		-1.5(6)
C(23)-C(24)-C(25)-C(26)		1.9(6)
C(24)-C(25)-C(26)-C(21)		-1.5(6)

are oriented away from the ring. Both compounds exhibit the *trans* orientation of the amide group and participate in intermolecular N – H ---- O hydrogen bonds.

The O(1) – C(1) – N(1) – C(11) torsion angle in compound (I) is 5.0(2)°, indicating that the amide group is essentially planar in this molecule. The benzene ring is sharply twisted out of the plane of the amide group; the C(16) – C(11) – N(1) – C(1) torsion angle is -22.7(2)°. This twist reduces π-interactions between the amide group and the benzene ring. In compound (II) the amide group is also planar (the O(1) – C(1) – N(1) – C(11) torsion angle is -1.1(6)°) and the benzene ring is twisted even more sharply out of the amide plane; the C(16) – C(11) – N(1)– C(1) torsion angle is -39.8(6)°. In compound (II) the C(22) – C(21) – C(1) – N(1) torsion angle is -31.2(6)° indicating that the second ring is also twisted out of the plane of the amide group. There is therefore little through-conjugation in compound (II). Figure 9.3 shows an edge on view of compound (II) and shows that the compound consists of three non-parallel planes: through the two benzene rings and the amide group.

The C = O bond lengths of 1.229(3) Å and 1.220(7) Å for compounds (I) and (II) respectively are similar (within experimental error) to the value of 1.219(3) Å found in acetanilide[45] and similar to the value of 1.223(6) Å and 1.232(3) Å found in *p*-hydroxyacetanilide[75,76]. The N(1) – C(11) bond lengths at 1.421(3) Å and 1.439(6) Å are longer than the corresponding value of 1.413(3) Å in acetanilide and the average value of 1.411(4) Å in the *p*-methoxy phosphoramidate anlogue. The C(1) – N(1) bond lengths of 1.352(3) Å and 1.362(7) Å are equal (within

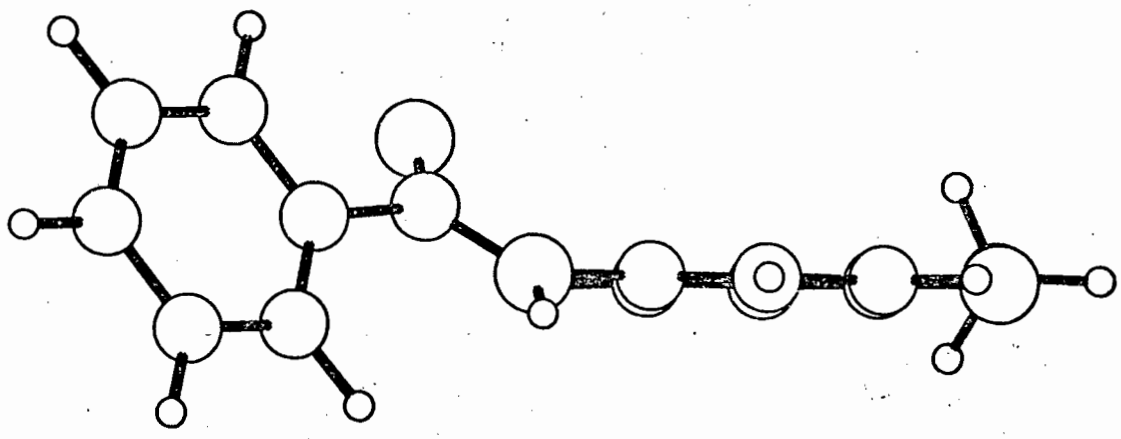


Figure 9.3 An edge-on view of compound (II)

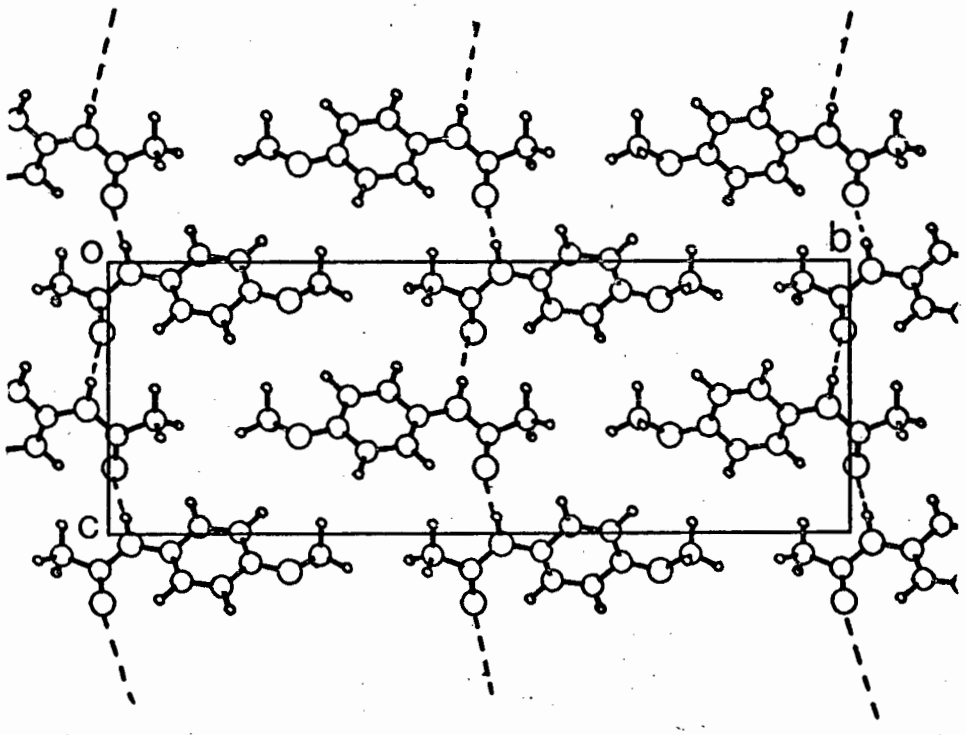


Figure 9.4 A view of the hydrogen bonding in compound (I)

experimental error) to the value of 1.354(3) Å in acetanilide. The N(1) – C(11) bond is expected to show considerable double-bond character and the lengthening in the two title compounds indicates that there is only negligible π-interaction between the ring and the amide group. This lengthening on *p*-substitution has also been found in *p*-chloroacetanilide[77], *p*-bromoacetanilide[78] and *p*-methylacetanilide[79] where interaction of the *p*-substituent with the ring is expected to enhance any π-interaction of the amide group with the ring.

The Benzene Ring Geometry

The pattern of distortions in the benzene rings in both molecules is indicative of the interaction of the substituent with the ring. The *ipso* angles, α_{OCH_3}, of the *p*-methoxy substituents of the rings, C(13) – C(14) – C(15), are 119.9(2)° and 122.0(5)° for compounds (I) and (II) respectively. The value for compound (I) is identical with the mean value of 119.9(1)° derived from many structural results on *p*-substituted anisoles[48] and is in agreement with the values of 119.3(3)° and 118.7(3)° found in the *p*-methoxy phosphoramidate analogue discussed previously. On the other hand the value of α_{OCH_3} for compound (II) is much larger than the mean value of 119.9(1)°. For a substituent capable of sharing π-electrons with the ring α increases with increasing conjugation[48]. This affect may also be attributed to through-conjugation. The large value of α_{OCH_3} in compound (II) indicates that the interaction of the methoxy substituent with the ring is more pronounced than in compound (I).

The value of α_{NH} (C(16) -- C(11) -- C(12)) in compound (II) is 120.7(5)° in comparison with the standard value of 119.2(2)° determined for many

derivatives of aniline (Ph -- NH -- X)[50] and is larger than the value of 120.0(2)° found in 3-pyridylcarboxanilide[60]. The enlarged value of α_{NH} in compound (II) is further evidence for through-conjugation in this compound. The α_{CONH-} angle of compound (II) (C(26) -- C(21) -- C(22), 121.6(5)°, is larger than the mean value of 119.4(2)° found for a number of benzamide derivatives (Ph – C(O) – NH – X)[80] and is larger than the value of 120.1(4)° found in hippuric acid (the largest value reported to date)[81]. The enlargement of this angle in compound (II) may also be ascribed to through-conjugation.

In compound (II) the bonds C(12) – C(13) and C(15) -- C(16) (mean = 1.410(8) Å) are longer than the other four bonds in the ring (mean = 1.372(8) Å). In the benzamide ring similar effects are found; the C(22) – C(23) and C(25) – C(26) bonds (mean = 1.416(8) Å) are longer than the other four (mean = 1.370(8) Å). Similar effects have been found in other *p*-substituted benzene derivatives and are a consequence of the lowering of the ideal D_{6h} (6/*mmm*) symmetry of the ring substitution[49,50,80]. In both compounds the benzene rings are planar (refer to Table 9.13). In compound (I) the amide and methoxy groups are bent out of the least-squares plane through the benzene ring and in compound (II) only the methoxy group is bent out of the plane of the ring. The C(14) -- O(2) bond length in compound (I), 1.379(3) Å, is shorter than that in compound (II), 1.409(6) Å. The value in compound (II) is longer than thosein the *p*-methoxy phosphoramidate analogue, 1.378(4) Å and 1.375(4) Å and the value of 1.356(3) Å found in *p*-methoxybenzoic acid[49].

Table 9.13 The Least-Squares Planes Calculated through the Benzene Rings and the Deviations of Selected Atoms from the Planes with e.s.d.'s in Parentheses.

Equations of the planes:

Plane 1: through C(11),C(12),C(13),C(14),C(15) and C(16) of compound (I).

$5.989x + 2.450y + 5.647z = 1.489$

Plane 2: through C(11),C(12),C(13),C(14),C(15) and C(16) of compound (II).

$10.081x + 2.942y - 6.347z = 1.319$

Plane 3: through C(21),C(22),C(23),C(24),C(25) and C(26) of compound (II).

$-5.608x + 2.807y + 6.831z = 3.207$

Deviations (Å) :

	Plane 1	Plane 2	Plane 3
N(1)	0.054(3)	-0.005(8)	-0.573(8)
C(11)	0.019(3)*	0.015(8)*	-0.680(8)
C(12)	-0.011(3)*	-0.009(8)*	-1.834(8)
C(13)	-0.008(3)*	0.000(8)*	-1.977(8)
C(14)	0.019(3)*	0.004(8)*	-0.944(8)
C(15)	-0.011(3)*	0.001(8)*	0.223(8)
C(16)	-0.008(3)*	-0.010(8)*	0.359(8)
O(1)	0.824(3)	1.398(8)	0.540(8)
C(1)	0.481(3)	0.678(8)	0.037(8)
C(2)	0.454(3)	0.137(8)	-0.173(8)
O(2)	0.071(3)	0.025(8)	-1.181(8)
C(21)		0.533(8)	-0.001(8)*
C(22)		-0.593(8)	0.000(8)*
C(23)		-0.674(8)	-0.003(8)*
C(24)		0.405(8)	0.007(8)*
C(25)		1.538(8)	-0.008(8)*
C(26)		1.612(8)	0.004(8)*

*Atoms used to calculate the least-squares plane

Hydrogen Bonding and Crystal Packing

Perspective views of the hydrogen bonding schemes in both compounds are given in Figures 9.1, 9.4 and 9.5. Details of the hydrogen bonding are listed in Table 9.14.

Both compounds participate in an intermolecular network of N — H --- O hydrogen bonds. The hydrogen bonding motifs of amides are well documented and in *trans* amides: glide, 2_1-axis and translational motifs are found[62,63,67]. In compound (I) a 2_1-axis motif is found in which molecules related by the 2_1 axis generate a network of N --- O hydrogen bonds extending parallel to the *b* axis. The glide or 2_1-axis motif gives rise to three modes labelled (a), (b) and (c) in Figure 9.6. In (a) the C = O --- N system is non linear and this situation closely matches the hydrogen bonding scheme in compound (I). Similar schemes are found in N-bromoacetamide[82], acetanilide[45], *p*-bromo-acetanilide[78] and *p*-chloroacetanilide[77]. The alternative translational motif is shown as (d) in Figure 9.6 and this motif occurs in compound (II). In this case the C = O --- N system is close to linearity and the translational axis is the *b* axis and is 5.252(3) Å long. In compound (I) the 2_1 axis length is 9.227(4) Å. The idealized motifs represented in Figure 9.6 require a 2_1-axis length of approximately 9 Å and a translational axis length of 5 Å if the hydrogen bonding requirements are to be met[62]. It is significant that in both compounds(I) and (II) the axes along which the hydrogen bonding extends closely match the expected lengths.

Molecules which contain bulky R_C groups tend to pack in the glide or 2_1-motif to permit the bulky groups to be sufficiently well separated

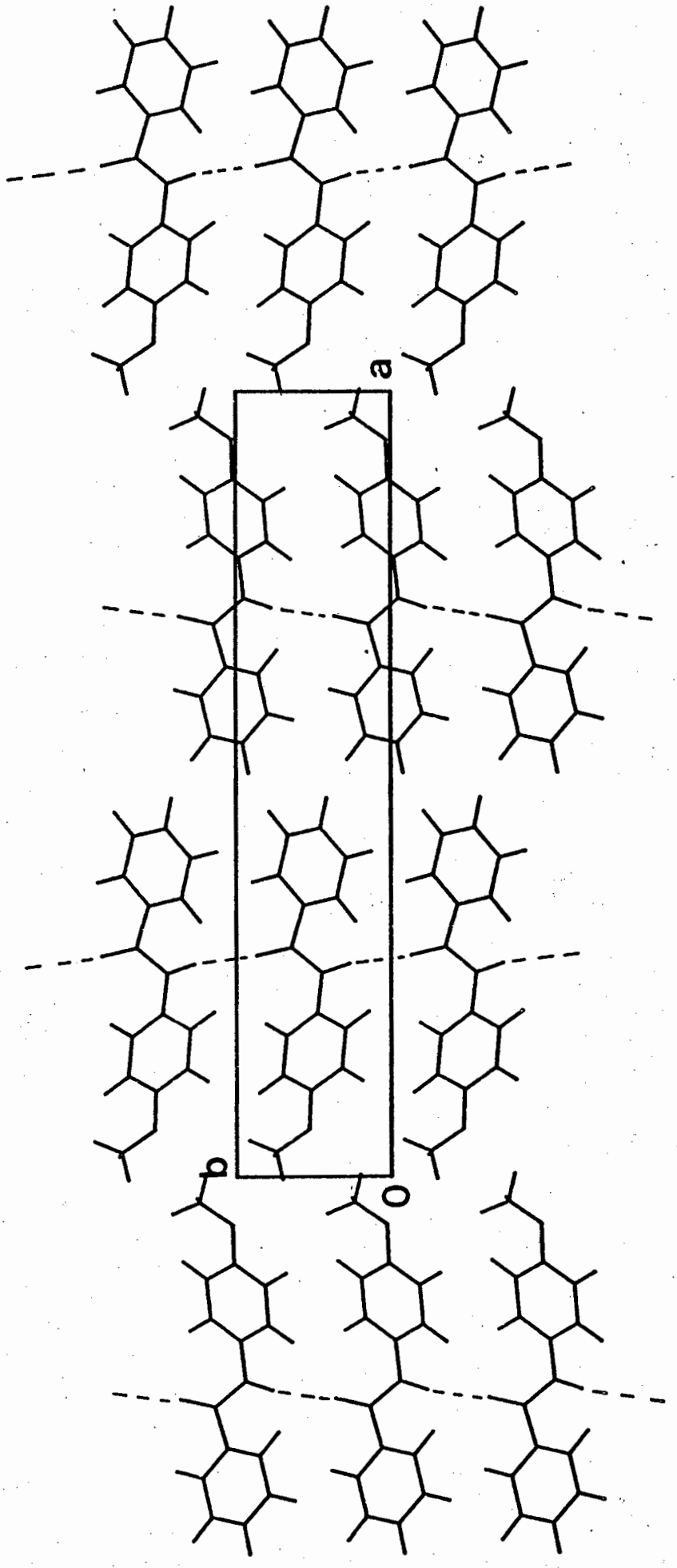


Figure 9.5 Hydrogen bonding in compound (II)

Table 9.14 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen bonding	Compound (I)	Compound (II)
N(1)-H(1) Å	0.99(1)	0.99(1)
H(1).....O(1) Å	1.88(1) [1]	2.21(1) [2]
N(1).....O(1) Å	2.875(5) [1]	3.105(7) [2]
N(1)-H(1)-O(1)°	177.9(9)	169.3(9)

Close contacts	
Compound (I):	
O(11).....C(12)	3.420(5) [3]
Compound (II):	
C(22).....O(1)	3.476(8) [4]

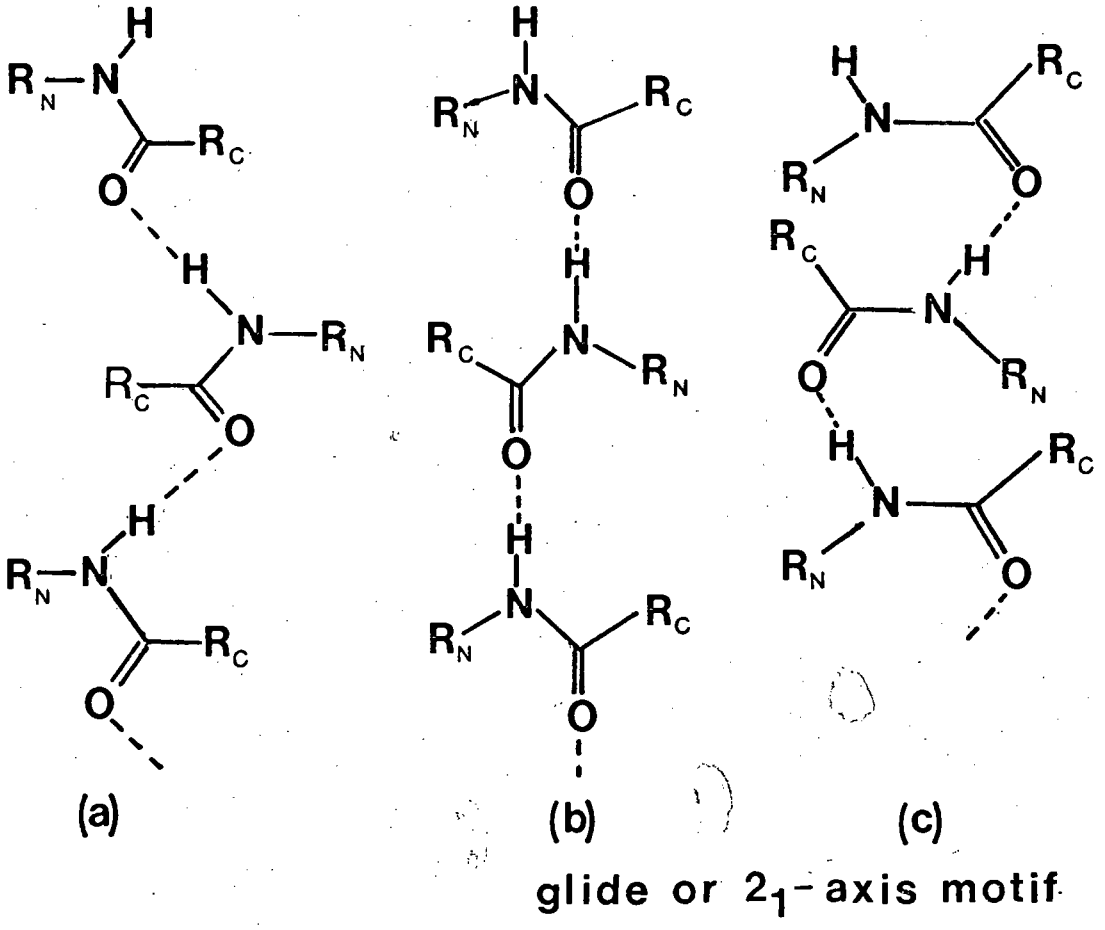
Symmetry codes:

[1] 0.5-x -y -0.5+z

[2] x 1+y z

[3] -0.5+x y 0.5-z

[4] X -1+Y Z



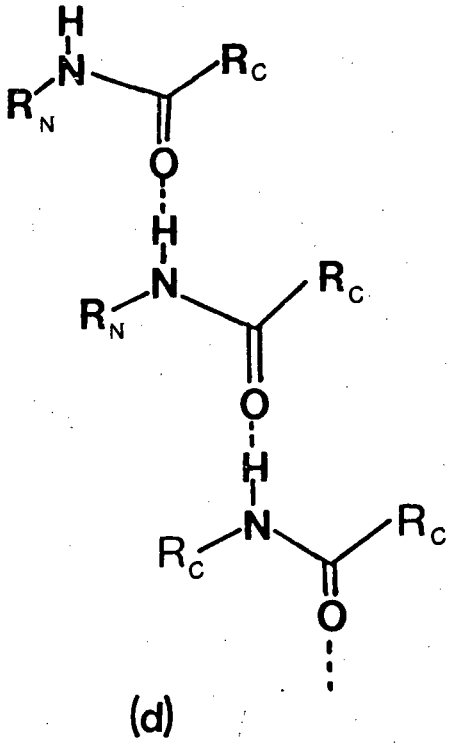


Figure 9.6 Hydrogen bonding motifs in amides

(for example in compound (I)). The translational motif, (d) embodies a close-packed arrangement and is suited to compound (II) since the benzene rings are able to overlap each other to form a layer type structure. A similar layer structure occurs in N-methyl-α-chloro-acetamide within the layer motif[83].

The hydrogen bonding motif has been shown to influence the twist of the benzene ring with respect to the amide group.[62] For example in N-methylbenzamide[62] the twist of the benzene ring with respect to the amide group is 14° as compared with the 24.6° angle of twist in benzamide[84]. The difference in the two angles is explained in terms of their different hydrogen bonding patterns. The hydrogen bonded molecules of N-methylbenzamide are related by a 2_1 axis; thus the bulky phenyl groups do not interfere with each other along the 2_1 axis; rather the phenyl group makes contact with the comparatively small $N - CH_3$ group. Benzamide[84] however is hydrogen bonded along a 5 Å axis which containing phenyl groups would experience unfavourable close contacts but for the 24° twist of the phenyl group out of the plane of the amide system. Analogous situations arise in the two title compounds. In compound (I) the hydrogen bonding motif (2_1-axis) permits close contact between the phenyl group and the relatively small methyl group at C(21); the twist of the phenyl ring out of the plane of the amide group is about 23°. In compound (II) which exhibits the translational motif the twist of the N(phenyl) group out of the plane of the amide group is increased to about 31°. This twist reduces lateral interactions between the bulky groups along the translational axis and permits overlap of the benzene rings as mentioned earlier.

The N ---- O hydrogen bond distance in compound (I) (2.875(5) Å) is close to the average value of 2.85 Å for *trans* secondary amides[62]. The value of 3.105(7) Å in compound (II) indicates that the hydrogen bond in this compound is fairly weak[54]. It appears that the length of the N ---- O bond in secondary amides does not depend on the mode of hydrogen bonding nor on the *cis* or *trans* configuration of the amide group.[62]

Further views of the crystal packing in both title compounds are given in Figures 9.7 and 9.8. The ribbon-like layer packing in compound (I) is evident. The individual ribbon layers are held together by van der Waals interactions along the *b* axis with hydrogen bonds holding the layers together along the *c* axis. Compound (II) packs in a zig-zag layer type motif with layer overlap perpendicular to the hydrogen bonding axis.

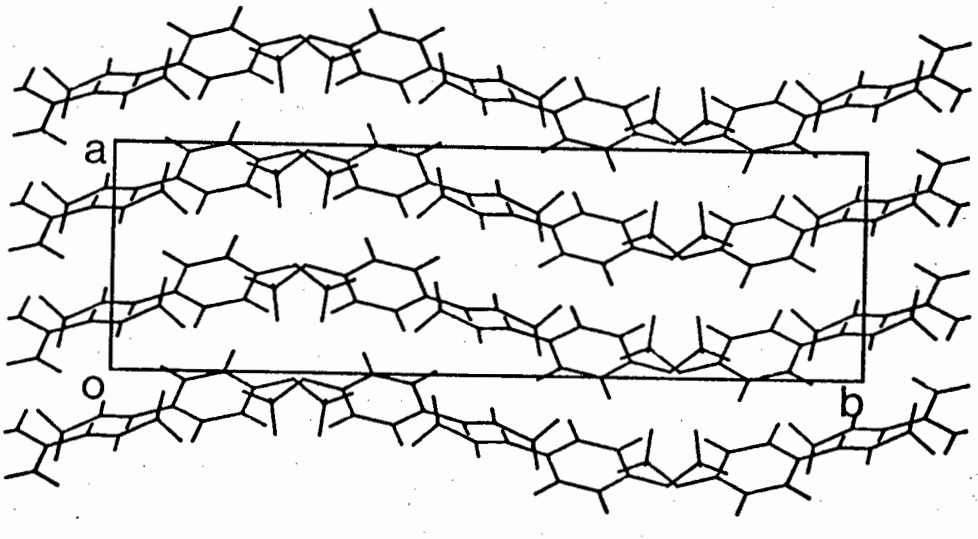
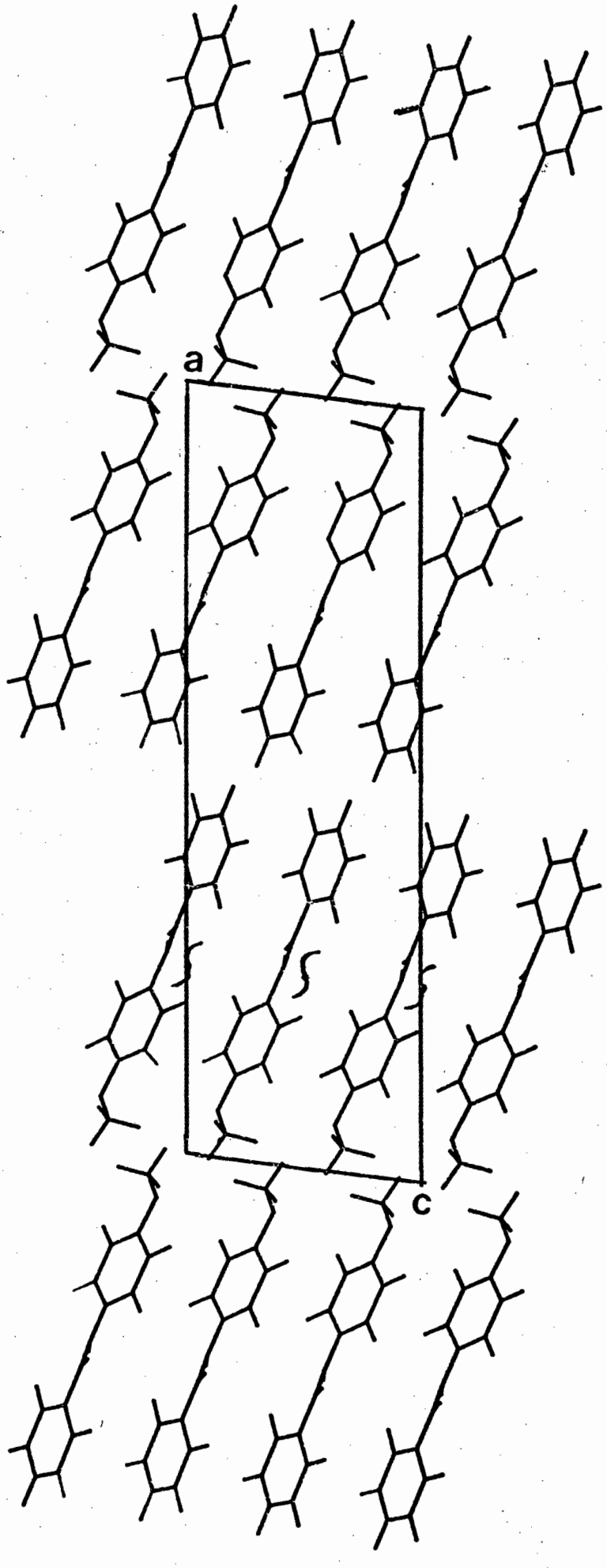


Figure 9.7 Crystal packing in compound (I)


a
c

CHAPTER TEN

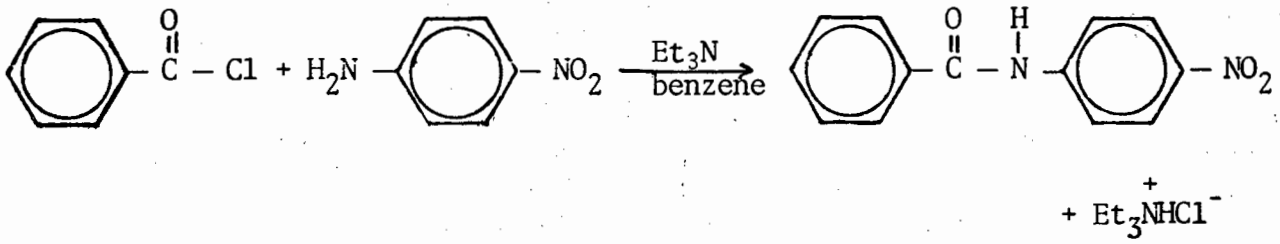
10. THE CRYSTAL AND MOLECULAR STRUCTURE OF BENZ-*p*-NITROANILIDE

10.1 Introduction

Originally it was hoped to determine the crystal structure of *p*-nitroacetanilide as the carboxylic analogue of the dimethyl-N-(*p*-nitrophenyl) phosphoramidate. However we were unable to obtain suitable crystals and so the crystal structure of benz-*p*-nitroanilide was determined.

10.2 Experimental

The title compound was synthesized according to the reaction:



The method of synthesis is described in the previous chapter. Found for $C_{13}H_{10}N_2O_3$: C = 64.56%, H = 4.31%, N = 11.75%. Calculated C = 64.46%, H = 4.13%, N = 11.57%. Suitable crystals were obtained by recrystallizing from methanol. Preliminary oscillation and Weissenberg photography ($0kl$, $1kl$, $h0l$, $h1l$, $hk0$ and $hk1$) indicated a triclinic space group. The structure was subsequently solved in the centrosymmetric space group $P\bar{1}$ [11]. Details of the crystal data and the diffractometer data are given in Table 10.1 No absorption corrections

Table 10.1 Crystal Data and Experimental and Refinement Parameters for Structure Analysis

Crystal data	
Molecular formula	$C_{13}H_{10}N_2O_3$
M_r	242.3
Space group	$P\bar{1}$
a	13.444(7) Å
b	5.334(3) Å
c	8.074(4) Å
α	91.93(2)°
β	103.09(2)°
γ	97.31(2)°
V	558.3 Å^3
D_m	1.43 Mgm^{-3}
D_c	1.44 (for Z = 2) Mgm^{-3}
$\mu(MoK_\alpha)$	0.044 mm^{-1}
μR_{min}, μR_{max}	0.002, 0.014
A*	1.0
F(000)	254
Data collection	
Crystal dimensions	0.65 x 0.3 x 0.1 mm
Scan mode	$\omega - 2\theta$
Scan width	1.4° @
Scan speed	0.05° @ s^{-1}
Range scanned (2θ)	4 - 46°
Stability of standard reflections	<1 %
Number of reflections collected	1492
Number of 'observed' reflections	953 with $I_{rel} > 2\sigma I_{rel}$
Number of variables	171
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.051
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.042
Weighting scheme w	$(\sigma^2 F)^{-1}$
U_{iso}(aromatic H)	0.062(4) Å^2
U_{iso}(amide H)	0.07(2) Å^2

were applied to the data. The density was determined in a density column.[39]

10.3 Solution and Refinement of the Structure

Initially some difficulty in solving the structure was encountered using the automatic direct methods routine of the SHELX[12] program system. A large number of *E*-maps were calculated (20 in total) and the 20th map yielded a chemically meaningful structure in which about ¾ of the heavy atoms were located. The remaining heavy atoms were located in a difference electron-density map calculated after three cycles of least-squares refinement. Most of the hydrogen atoms, including the amide hydrogen atom were located in a difference electron-density map calculated after further cycles of least-squares refinement. The final refinements were carried out with the heavy atoms treated anisotropically, with the methyl hydrogen atoms refined as a rigid group and the aromatic hydrogen atoms constrained at 1.08 Å from their carbon atoms, their positions dictated by the geometry of the molecule. The amide hydrogen atom was constrained at 1.00 ± 0.01 Å from the nitrogen atom. The isotropic temperature factors of the methyl, aromatic and amide hydrogen atoms were treated as three single parameters. In the final cycle of least-squares refinement the average parameter to error shift was less than 1.5 %. A difference electron-density map calculated after the final refinements revealed no peaks > 0.1 e·Å^{-3}. An analysis of variance calculated after the final refinement is given in Table 10.2. Details of the final refinement are given in Table 10.1 and lists of observed and calculated structure factors in Appendix 10. The final atomic coordinates and anisotropic temperature factors are listed in Tables 10.3, 10.4 and 10.5 respectively.

TABLE 10.2. Analysis of Variance[a]

(a) By parity group

	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	127	126	123	121	108	104	124	120	953
V	61	60	54	48	50	51	52	64	55

(b) As a function of sin (θ)

sin(θ)	0	.17	.21	.24	.27	.29	.32	.34	.35	.38	.40
N	117	93	97	96	85	132	95	47	137	54	
V	86	62	55	49	42	45	46	39	48	57	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.18	.20	.22	.23	.26	.28	.31	.35	.41	1.00
N	117	115	110	54	116	81	97	83	90	90	
V	51	42	50	40	45	49	53	60	57	93	

(d) As a function of |Miller Index|

$\lvert h \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	52	95	98	100	106	95	86	71	67	57	41	31	25	22	7
V	55	67	66	56	51	53	53	59	45	51	55	40	53	44	31

$\lvert k \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	117	256	227	171	121	61	0	0	0	0	0	0	0	0	0
V	62	59	58	50	47	49	0	0	0	0	0	0	0	0	0

$\lvert l \rvert$	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	90	180	181	139	141	94	68	43	17	0	0	0	0	0	0
V	65	59	62	55	50	49	41	43	32	0	0	0	0	0	0

[a] N = Number of reflections in the group; $V = 100[M\Sigma(w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table 10.3 Fractional Atomic Coordinates ($x10^4$) of the Heavy Atoms with e.s.d.'s in Parentheses

	x	y	z
O(1)	5024(2)	-1157(5)	2577(4)
O(2)	9704(3)	6348(8)	7645(5)
O(3)	10004(3)	2998(7)	6366(5)
N(1)	5338(2)	3168(6)	2736(4)
N(2)	9439(3)	4533(10)	6595(6)
C(1)	4716(3)	887(8)	2242(5)
C(11)	6378(3)	3453(8)	3674(5)
C(12)	6722(3)	5553(8)	4821(5)
C(13)	7737(3)	5940(8)	5776(5)
C(14)	8373(3)	4212(8)	5545(5)
C(15)	8059(3)	2135(8)	4394(6)
C(16)	7048(3)	1756(8)	3446(5)
C(21)	3641(3)	1050(7)	1304(5)
C(22)	3359(3)	3016(8)	284(5)
C(23)	2340(3)	2977(8)	-590(5)
C(24)	1596(3)	1003(9)	-444(6)
C(25)	1871(3)	-960(8)	565(6)
C(26)	2886(3)	-953(7)	1439(5)

Table 10.4 Fractional Atomic Coordinates ($x10^3$) of the Hydrogen Atoms.

	x	y	z
H(1)	499	470	267
H(121)	620	689	497
H(131)	802	756	668
H(151)	859	84	423
H(161)	679	16	253
H(221)	393	457	18
H(231)	213	449	-140
H(241)	83	101	-105
H(251)	129	-250	68
H(261)	310	-249	222

Table 10.5 Anisotropic Temperature Factors ($Å^2 \times 10^3$) of the Heavy Atoms with e.s.d.'s in Parentheses.

	U11	U22	U33	U23	U13	U12
O(1)	40(2)	27(2)	90(3)	14(2)	-6(2)	7(2)
O(2)	57(3)	122(4)	83(3)	-24(3)	-12(2)	-14(2)
O(3)	43(2)	92(3)	85(3)	32(2)	-1(2)	17(2)
N(1)	29(2)	24(2)	49(3)	1(2)	-5(2)	2(2)
N(2)	45(3)	80(4)	50(3)	20(3)	4(3)	-5(3)
C(1)	38(3)	33(3)	43(3)	5(2)	7(2)	5(2)
C(11)	34(3)	32(3)	38(3)	6(2)	7(2)	0(2)
C(12)	39(3)	34(3)	39(3)	2(2)	0(2)	3(2)
C(13)	48(3)	33(3)	42(3)	2(2)	6(3)	-6(2)
C(14)	28(3)	44(3)	40(3)	11(3)	-3(2)	-6(2)
C(15)	30(3)	43(3)	54(3)	13(3)	7(2)	6(2)
C(16)	38(3)	34(3)	49(3)	2(2)	9(3)	3(2)
C(21)	30(3)	29(3)	35(3)	2(2)	2(2)	3(2)
C(22)	42(3)	32(3)	41(3)	4(2)	5(2)	2(2)
C(23)	47(3)	38(3)	44(3)	9(2)	-2(3)	14(2)
C(24)	37(3)	47(3)	46(3)	1(3)	-1(3)	5(3)
C(25)	38(3)	40(3)	52(3)	4(2)	2(3)	-4(2)
C(26)	38(3)	30(3)	44(3)	4(2)	1(2)	1(2)

10.4 Description of the Structure and Discussion

A perspective view of the title compound showing the atomic nomenclature is shown in Figure 10.1. Principal bond lengths, angles and torsion angles are listed in Tables 10.6, 10.7 and 10.8 respectively.

The conformation of the title compound is similar to that observed in the *p*-methoxy analogue discussed earlier. The compound crystallizes in the *endo* conformation in which the C = O group is *cis* to the benzene ring. The compound participates in a network of N – H --- O intermolecular hydrogen bonds. The molecule is non-planar and may be divided into three non-parallel planes, through benzene ring one, benzene ring two and the amide group. This may be seen from the side view of the molecule shown in Figure 10.2.

The amide group is planar; the O(1) – C(1) – N(1) – C(11) torsion angle is 0(4)°. Once again the benzene rings are sharply twisted out of the plane of the amide group. The C(1) – N(1) – C(11) – C(16) torsion angle is 35.5(4)° and the N(1) – C(1) – C(21) – C(22) torsion angle is 29.4(4)°. The C = O bond length in the title compound is 1.231(4) Å and this is equal (in terms of the estimated standard deviations) to the value of 1.229(3) Å found in the *p*-nitrobenzanilide. The N – C(1) bond, 1.376(5) Å, is longer (i.e. has more single bond character) than the corresponding bond in acetanilide[45], 1.354(3) Å, and in the *p*-methoxybenzanilide analogue. The N – C(11) bond, 1.418(5) Å, is comparable in length to the value of 1.413(3) Å found in acetanilide[45].

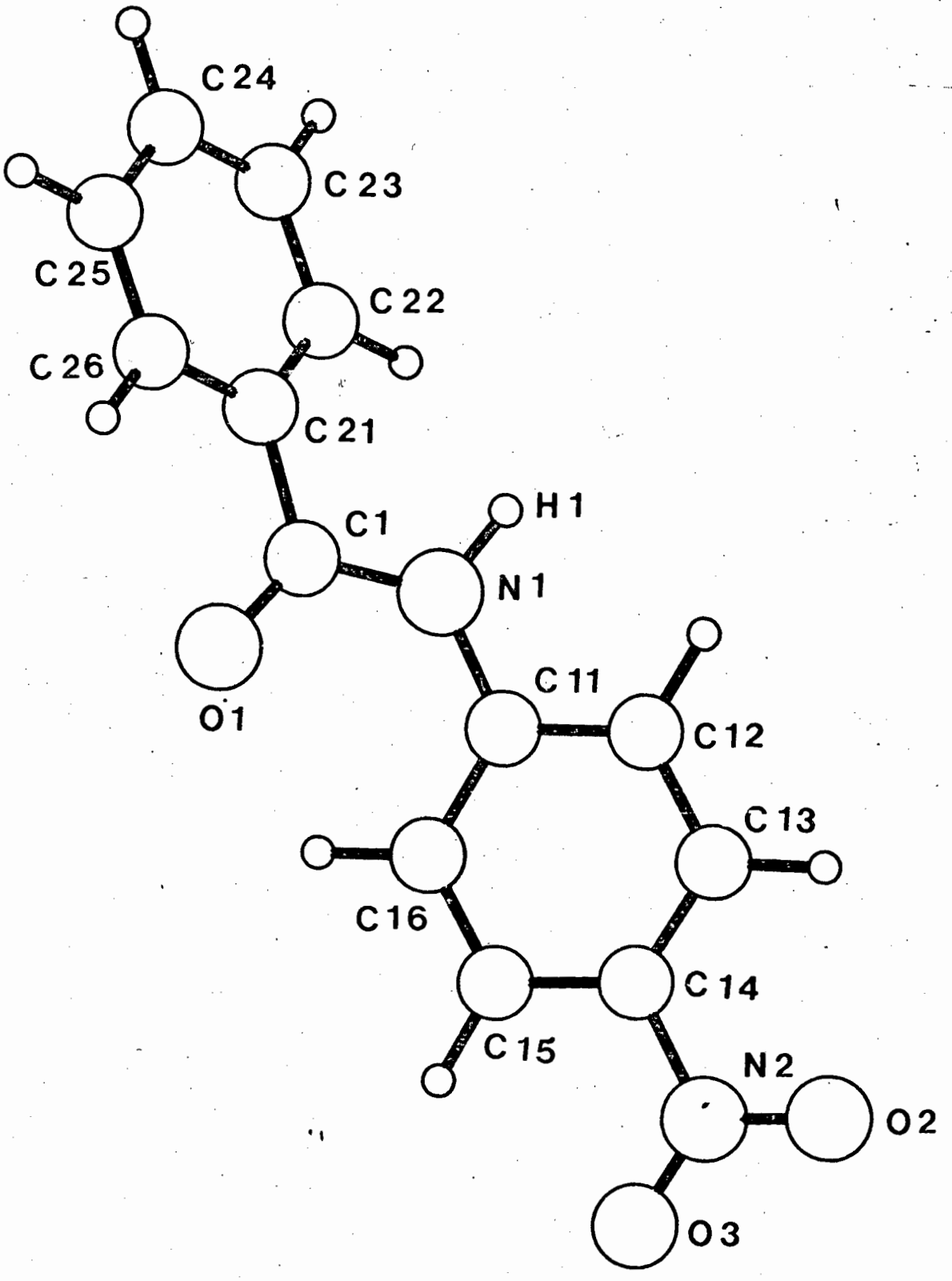


Figure 10.1 A perspective view of the title compound

Table 10.6 Principal Bond Lengths (Å) with e.s.d.'s in Parentheses.

C(1)-O(1)	1.231(4)
N(2)-O(2)	1.226(5)
N(2)-O(3)	1.223(5)
C(1)-N(1)	1.376(5)
C(11)-N(1)	1.418(5)
C(14)-N(2)	1.476(5)
C(21)-C(1)	1.486(5)
C(12)-C(11)	1.389(5)
C(16)-C(11)	1.391(5)
C(13)-C(12)	1.393(5)
C(14)-C(13)	1.370(5)
C(15)-C(14)	1.380(5)
C(16)-C(15)	1.387(5)
C(22)-C(21)	1.393(5)
C(26)-C(21)	1.402(5)
C(23)-C(22)	1.389(5)
C(24)-C(23)	1.385(6)
C(25)-C(24)	1.385(5)
C(26)-C(25)	1.387(5)

Table 10.7 Principal Bond Angles (°) with e.s.d.'s in Parentheses.

C(11)-N(1)-C(1)	125.0(4)
O(3)-N(2)-O(2)	123.9(5)
C(14)-N(2)-O(2)	117.5(5)
C(14)-N(2)-O(3)	118.6(5)
N(1)-C(1)-O(1)	122.3(4)
C(21)-C(1)-O(1)	122.0(4)
C(21)-C(1)-N(1)	115.6(4)
C(12)-C(11)-N(1)	117.3(4)
C(16)-C(11)-N(1)	122.3(4)
C(16)-C(11)-C(12)	120.4(4)
C(13)-C(12)-C(11)	120.0(4)
C(14)-C(13)-C(12)	118.2(4)
C(13)-C(14)-N(2)	119.1(4)
C(15)-C(14)-N(2)	117.8(4)
C(15)-C(14)-C(13)	123.1(4)
C(16)-C(15)-C(14)	118.5(4)
C(15)-C(16)-C(11)	119.8(4)
C(22)-C(21)-C(1)	123.9(4)
C(26)-C(21)-C(1)	116.8(4)
C(26)-C(21)-C(22)	119.2(4)
C(23)-C(22)-C(21)	120.1(4)
C(24)-C(23)-C(22)	120.4(4)
C(25)-C(24)-C(23)	119.8(4)
C(26)-C(25)-C(24)	120.4(4)
C(25)-C(26)-C(21)	120.4(4)

Table 10.8 Selected Torsion Angles (°) with e.s.d.'s in Parentheses.

C(11)-N(1)-C(1)-O(1)	0.0(4)
C(11)-N(1)-C(1)-C(21)	178.5(4)
C(1)-N(1)-C(11)-C(16)	35.5(4)
O(2)-N(2)-C(14)-C(13)	0.0(4)
O(2)-N(2)-C(14)-C(15)	-178.5(4)
O(3)-N(2)-C(14)-C(13)	-179.1(4)
O(3)-N(2)-C(14)-C(15)	2.2(4)
O(1)-C(1)-C(21)-C(22)	-151.7(4)
O(1)-C(1)-C(21)-C(26)	26.5(4)
N(1)-C(1)-C(21)-C(22)	29.4(4)
N(1)-C(1)-C(21)-C(26)	-152.4(4)
N(1)-C(11)-C(12)-C(13)	180.0(4)
C(16)-C(11)-C(12)-C(13)	-1.4(4)
N(1)-C(11)-C(16)-C(15)	180.0(4)
C(12)-C(11)-C(16)-C(15)	1.4(4)
C(11)-C(12)-C(13)-C(14)	0.0(4)
C(12)-C(13)-C(14)-N(2)	-177.8(4)
C(12)-C(13)-C(14)-C(15)	1.0(4)
N(2)-C(14)-C(15)-C(16)	177.5(4)
C(13)-C(14)-C(15)-C(16)	-1.1(4)
C(14)-C(15)-C(16)-C(11)	0.0(4)
C(1)-C(21)-C(22)-C(23)	178.0(4)
C(26)-C(21)-C(22)-C(23)	0.0(4)
C(1)-C(21)-C(26)-C(25)	-178.4(4)
C(22)-C(21)-C(26)-C(25)	0.0(4)
C(21)-C(22)-C(23)-C(24)	0.0(4)
C(22)-C(23)-C(24)-C(25)	0.0(4)
C(23)-C(24)-C(25)-C(26)	0.0(4)
C(24)-C(25)-C(26)-C(21)	0.0(4)

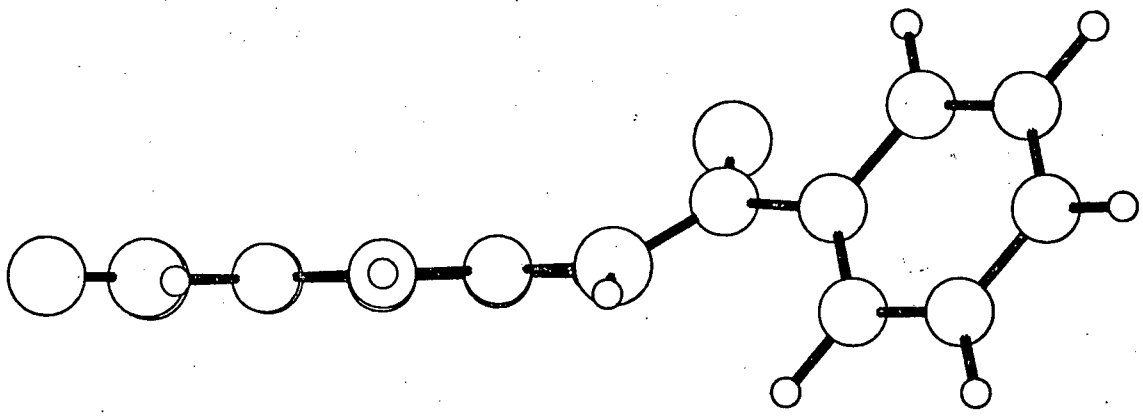


Figure 10.2 An edge-on view of the title compound

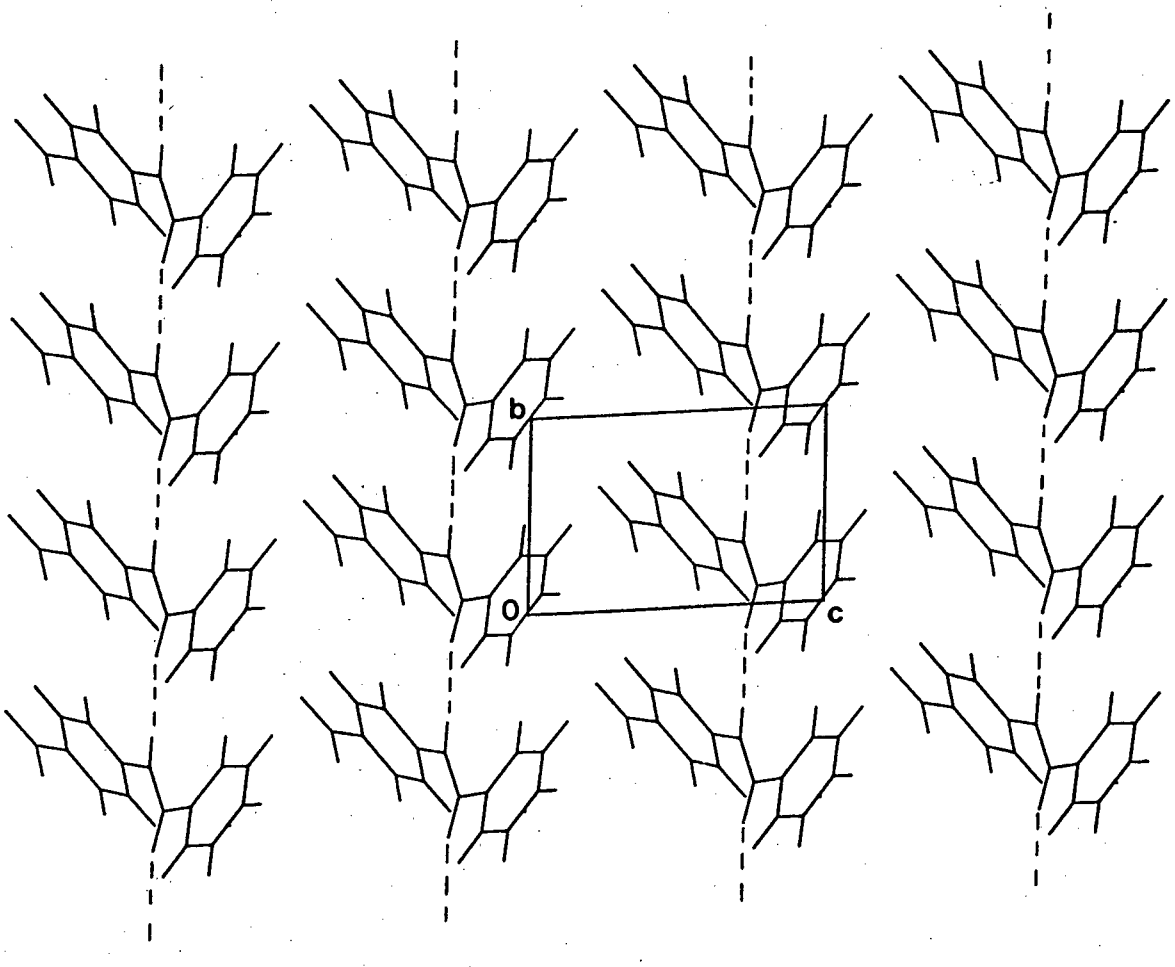


Figure 10.3 Hydrogen bonding in the title compound

The Benzene Ring Geometry

The pattern of distortions in the benzene ring geometry matches that expected with the substituents present. The deviations of selected atoms from the least-squares planes through the six-membered rings are listed in Table 10.9. The *ipso* angle of the nitro group, α_{NO_2} (C(13) – C(14) – C(51)), is 123.1(4)° and is greater than the average value of 122.1(1)° obtained for a large number of derivatives of nitrobenzene[46]. The value of α_{NO_2} in the title compound is smaller than the value of 124.7(10)° found in the *p*-nitro phosphoramidate analogue discussed earlier but is similar to the value of 123.3(1)° found in *p*-nitrobenzoic acid[57] and 123.4(1)° in *p*-dinitrobenzene[58]. The large value of α_{NO_2} in the title compound is however consistent with the strong σ-withdrawing character of the nitro substituent[46].

The amide *ipso* angle α_{NH} (C(16) – C(11) – C(12)), 120.4(4)°, is larger than the average value of 119.2(2)° found in aniline derivatives[50] and is close to the value of 120.3(9)° found in the *p*-nitrophosphoramidate analogue. The larger value of α_{NH} may be as a result of through-conjugation[50] or as a result of the influence of the nitro substituent on this angle[50]. The α_{CONH} angle (C(26) – C(21) – C(22)) is 119.2(4)° which is in agreement with the average value of 119.4(2)° found for a number of benzamide derivatives[80].

The distortion of the symmetry of the ring attached to the nitrogen atom principally involves the half of the ring closest to the nitro group. The two bonds involving C(14), C(13) – C(14) and C(15) – C(14), are shorter than the other four bonds. In both rings, most of the C – C bonds are shorter than the accepted average of 1.397 – 1.399 Å for the C – C bond

Table 10.9 The Least-Squares Planes Calculated through the Benzene Rings and the Deviations of Selected Atoms from the Planes with e.s.d.'s in Parentheses.

Equations of the planes:

Plane 1 : Through C(11),C(12),C(13),C(15) and C(16)

$-5.128x - 2.836y + 6.613z = -1.829$

Plane 2 : Through C(21),C(22),C(23),C(24),C(25) and C(26)

$-5.810x + 2.653y + 6.715z = -0.962$

Deviations (Å) :

	Plane 1	Plane 2
N(1)	0.003(5)	0.538(5)
C(11)	0.009(5)*	0.639(5)
C(12)	-0.005(5)*	1.767(5)
C(13)	-0.003(5)*	1.922(5)
C(14)	0.008(5)*	0.938(5)
C(15)	-0.004(5)*	-0.204(5)
C(16)	-0.005(5)*	-0.353(5)
C(21)	0.527(5)	0.007(5)*
C(22)	-0.561(5)	0.002(5)*
C(23)	-0.605(5)	-0.004(5)*
C(24)	0.423(5)	0.003(5)*
C(25)	1.516(5)	0.000(5)*
C(26)	1.571(5)	-0.002(5)*
N(2)	0.065(5)	1.109(5)
C(1)	0.642(5)	-0.037(5)
O(1)	1.285(5)	-0.534(5)

*Atoms used in the calculation of the least-squares plane

length in benzene[57,85,86]. These affects have been observed in many substituted benzene derivatives and have been found in virtually all the substituted benzene derivatives discussed in the previous chapters. The nitro group is coplanar (within experimental error) with the benzene ring to which it is attached. In all other respects the geometry of the nitro group is similar to that observed in other *p*-nitro substituted aromatic compounds[58].

Hydrogen Bonding and Crystal Packing

The title compound crystallizes in the *trans* orientation of the amide group and participates in an intermolecular network of N – H---- O = C hydrogen bonds. A perspective view of the hydrogen bonding scheme is shown in Figure 10.3 and details of the hydrogen bonding are listed in Table 10.10.

The hydrogen bonding motif is the translational type (refer to the previous chapter) in which molecules which participate in the hydrogen bonding are related by a translation axis (the *b* axis in this case) of 5.334(3) Å. This gives rise to a ribbon of hydrogen bonding extending parallel to the *b* axis. Characteristically, to reduce lateral contacts between the bulky aromatic groups along the translation axis the benzene rings are twisted out of the plane of the amide group. This twist permits layer overlap of the rings along *b* and hence close contact between related aromatic rings. This hydrogen bonding motif is almost identical to that found in the *p*-methoxy analogue discussed in the previous chapter.

An alternative view of the crystal packing is shown in Figure 10.4.

Table 10.10 Hydrogen Bonding Data and Close Contacts (Å) with e.s.d.'s in Parentheses.

Hydrogen bonding:	
N(1)-H(1)	0.99(1) Å
N(1).....O(1)	3.112(5) Å [1]
H(1).....O(1)	2.21(1) Å [1]
N(1)-H(1)-O(1)	151.2(9)°
Close contacts:	
C(12).....O(1)	3.307(5) [1]
O(2).....O(2)	3.373(5) [2]
C(24).....O(3)	3.258(5) [3]
O(3).....O(3)	3.121(5) [2]
N(2).....O(3)	2.964(5) [2]
C(14).....O(3)	3.195(5) [2]

Symmetry code:

[1] x 1+y z

[2] 2-x 1-y 1-z

[3] -1-x -y -1-z

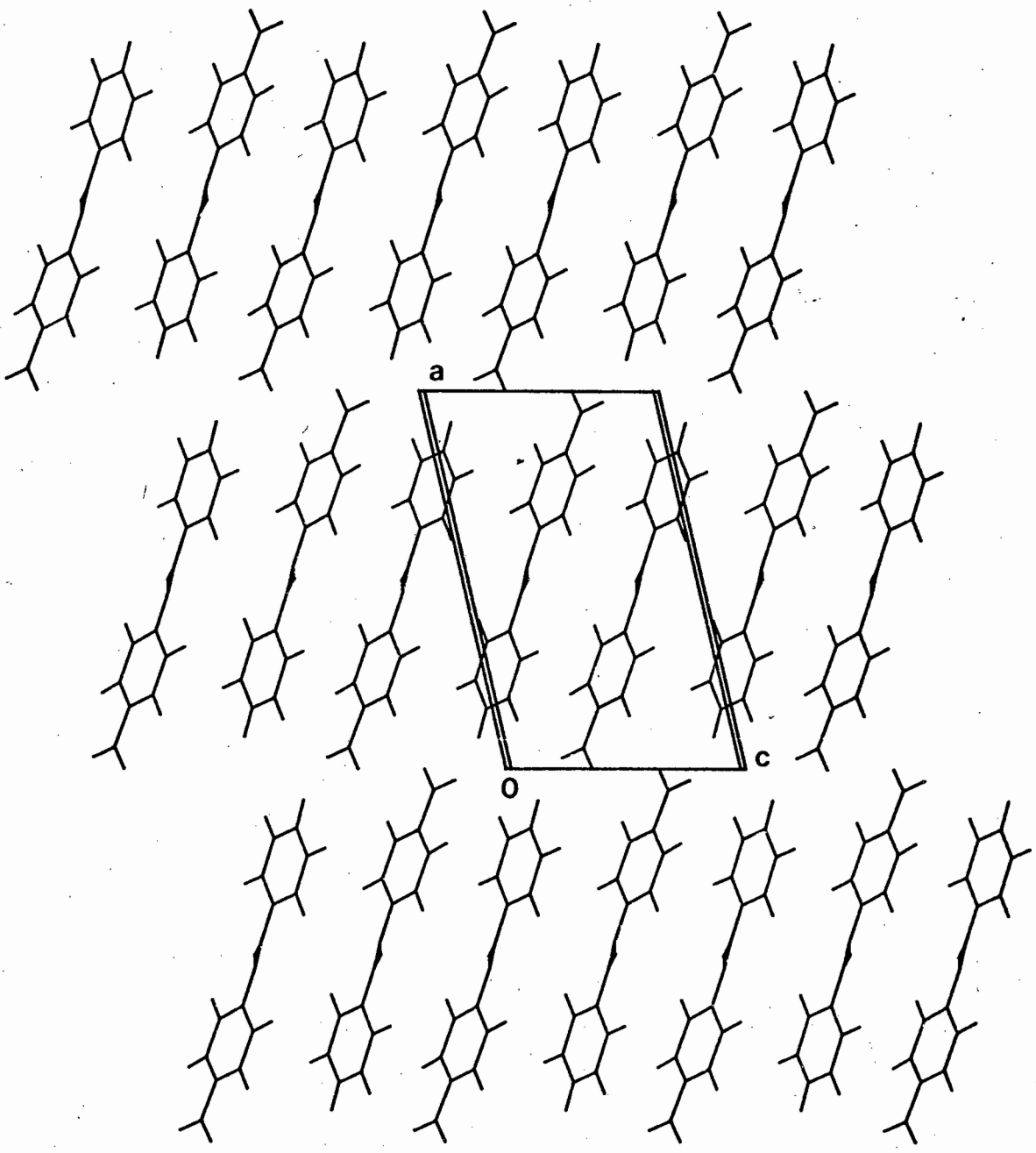


Figure 10.4 A view of the crystal packing

The compound packs in the well known[68] layer fashion of aromatic compounds (characterised by the short *b* axis of 5.334 Å). There are a number of close contacts (<3.5 Å); refer to Table 10.10 . These principally involve an interaction between the nitro group and the benzene rings of adjacent molecules. Similar interactions were noted in the *p*-nitro phosphoramidate analogue. The crystal structure of the *p*-methoxy analogue discussed in the previous chapter and that of the title compound are remarkably similar. Consider the space groups of the two compounds :

p-OCH_3: $P2_1/c$ a = 24.74, b = 5.252, c = 8.145 Å, β = 97°

p-NO_2 : $P\bar{1}$ a = 13.444, b = 5.334, c = 8.074 Å, α = 91, β = 103, γ = 97°

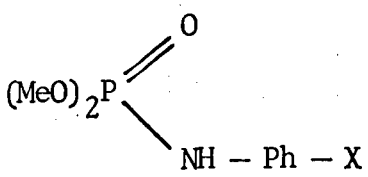
The *b* and *c* axes are almost the same and the *a* axis in the *p*-OCH_3 analogue is double that in the title compound. Both structures exhibit the translational hydrogen bonding motif along the *b* axis. Further comparisons between these two structures will be discussed in the next chapter.

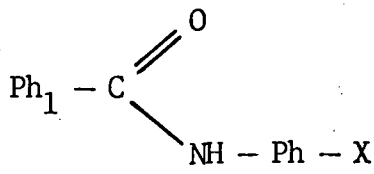
CHAPTER ELEVEN

11. STRUCTURAL CORRELATIONS ON CARBOXYLIC AND PHOSPHORIC AMIDES

11.1 Introduction

The crystal and molecular structures of a series of phosphoric (1-5) and carboxylic amides (6-8) have been discussed in the previous chapters. In this chapter the structural features of these compounds will be correlated with previously determined spectroscopic data in order to compare the bonding in phosphoric and carboxylic amides. In this chapter a large number of structures will be referred to, and in order to assist the reader these are shown in a fold out section at the end of this chapter.





(1) X = *p*-OMe
(2) = H
(3) = *p*-NO_2
(4) = *p*-Et
(5) = 2,6-diMe

(6) X = *p*-OMe
(7) = *p*-NO_2
(8) = *p*-OMe
Ph_1 = Me

SCHEME 11.1

11.2 Carboxylic Amides

The relevant structural parameters obtained for the carboxylic amides ((6)-(8)) are listed in Table 11.1, the data for N-methylbenzamide (9)[62] and acetanilide (10)[45] are included for comparative purposes.

Table 11.1 Structural Parameters in the Carboxylic Amides ((6)-(8)) with the Data for N-methylbenzamide[62] (9) and Acetamilide[45] (10)

Structure[a]	d(C=O)	d(C-N)	d(N-C_{ar})	τ_1[b]	τ_2[c]	τ_3[d]	∠(C-N-C_{ar})	α_X[e]	α_{NH}[e]	α_{CONH}[f]	$(\alpha_{CONH})_s$[g]	$(\alpha_X)_s$[g]
p-OMe benz (6)	1.220(7)	1.362(7)	1.439(6)	-1.1(6)	-39.8(6)	-31.2(6)	124.1(5)	122.0(5)	120.7	121.6(5)	119.4(2)	119.9(1)
N-methylbenz-amide[62] (9)	1.236(2)	1.328(2)	1.456(3)	12.1(2)	-	-13.5(2)	121.9(2)	-	-	118.9	119.4(2)	-
p-NO_2benz (7)	1.231(4)	1.376(5)	1.418(5)	0.0(4)	35.5(4)	29.4(4)	125.0(4)	123.1(4)	120.4(4)	119.2(4)	119.4(2)	122.1(1)
p-OMe acetanilide (8)	1.229(3)	1.352(3)	1.421(3)	5.0(2)	-22.7	-	127.2(2)	119.9(2)	119.0(2)	-	-	119.1(1)
Acetanilide[45] (10)	1.219(3)	1.354(3)	1.413(3)	-4.0(2)	20.0(2)	-	127.6(2)	-	119.6(2)	-	-	-

a All distances are in Å and angles in degrees

b τ_1 is the O(1) – C(1) – N(1) – C(11) torsion angle

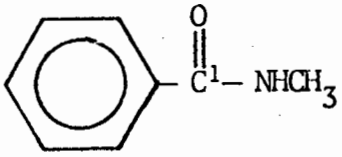
c τ_2 is the C(1) – N(1) – C(11) – C(16) torsion angle

d τ_3 is the N(1) – C(1) – C(21) – C(22) torsion angle

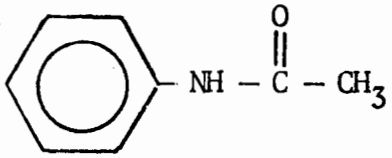
e α_X and α_{NH} are the *ipso* angles of the substituent (C(13) – C(14) – C(15)) and the amide group (C(16) – C(11) – C(12))

f α_{CONH} is the C(26) – C(21) – C(22) angle

g $(\alpha_X)_s$, $(\alpha_{NH})_x$ and $(\alpha_{CONH})_s$ are the standard values of the *ipso* angles above.



(9)

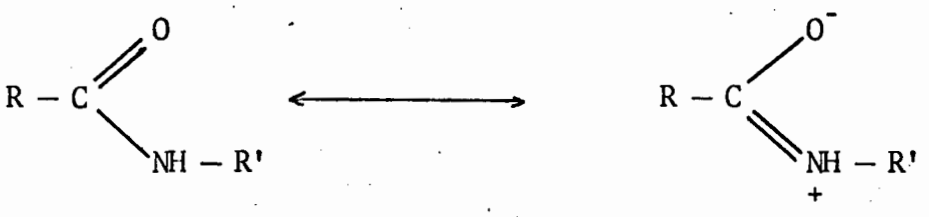


(10)

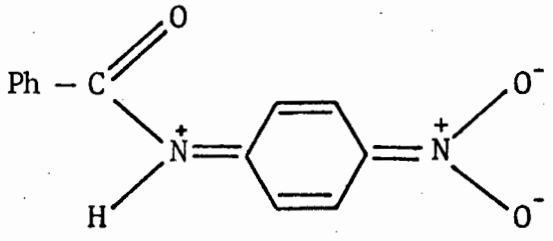
The partial double-bond character of the carbonyl-nitrogen bond requires the amide group to be planar. The resonance stabilization achieved in this way is large (the rotational barrier of the C – N bond for most amides is about 83 k J mole^{-1})[20] and consequently the achievement of a planar amide group is an important geometrical condition. In most of the amides listed in Table 11.1 the amide groups are close to planarity ($|\tau_1| \sim 5°$). Recent studies on simple peptides and other amides have however revealed that generally the amide group is non-planar and that the geometry of this group is distorted both by bulky side groups and by intermolecular contacts (especially hydrogen bonds)[33]. Variations in the detailed electron distribution within the amide function (as represented by the resonance structures (11) and (12) below) affect primarily the carbonyl-nitrogen bond order. In N-methyl-benzamide[62] the C(1) -- N bond is shortest, indicating a strong resonance interaction of the non-bonding electrons on nitrogen with the carbonyl group.

In the amides (6) to (8) the aromatic group competes with the carbonyl group for these non-bonding electrons and if the benzene ring is substituted with an electron-withdrawing substituent the resonance interaction of the non-bonding electrons with the carbonyl group is reduced and this is reflected in the lengthening of the C(1) – N bond. In the *p*-nitroanilide (7) the nitrogen lone pair is involved in conjugation with the *p*-nitro group (resonance structure 13), resulting in

the elongation of the C(1) – N bond and the shortening of the nitrogen-aromatic (N – C(11)) bond length. The relative contribution of (13) to the overall structure is reduced by the fact that the benzene ring and the amide group are not co-planar (τ_2 = 35.5(4)°).



(11) (12)



(13)

The π-interaction of the nitro group in resonance structure (13) has been discussed in terms of the effect of the substituent on the *ipso* angle α_{NO_2} and is evident from the large value of α_{NO_2} (123.1(4)°) in (7). In the *p*-OCH_3 substituted anilides, (6) and (8), the C(1) – N bond does not differ (within experimental error) from the length of this bond in acetanilide, while there is a lengthening of the N – Ar bond length over the value in acetanilide. In the benzanilide (6) the resonance interactions between the methoxy group and the nitrogen atoms (resulting in a shorter N – C_{Ar} bond) are reduced by the sharp twist (τ_2) of the benzene ring to the amide group. In the *p*-methoxy-acetanilide (8) both the NHAc and OMe substituents are resonance donors

with respect to the aromatic ring and direct conjugation effects between these two groups are minimised[87]. The reduced through-conjugation effect in compound (8) compared to compound (6) is evident from the values of α_{OCH_3} in these two compounds. In (6) α_{OCH_3} is larger than the standard value, while in compound (6) α_{OCH_3} is identical with the standard value of this angle. It is known that through-conjugation effects are responsible for the enlargement of α over the standard value[50] and this effect has been discussed in previous chapters.

In all of the compounds in Table 11.1 the C = O bond lengths remain relatively unaffected by the structural modifications. This is in contrast to the IR data on related substituted acetanilides where the C = O IR stretching frequencies are sensitive to the nature of the substituent on the benzene ring[35]. It must be realized however that all the compounds in Table 11.1 participate in C = O --- H — N hydrogen bonds and this has a significant influence on the C = O bond order (and length)[88]. In contrast however the IR data were collected in solution at low concentration where hydrogen bonding effects are small.

In all the amides listed in Table 11.1 the benzene rings are twisted out of the plane of the amide group, the deviation from planarity for benzanilides being greater than that for the acetanilides. This twist greatly reduces any resonance interaction between the rings and the amide group. It appears that the twist of the aromatic groups is a direct consequence of the hydrogen bonding motif adopted by these compounds and the twist out of planarity is greatest where bulky groups are involved (e.g. in the benzanilides, (6) and (7)). It was shown (see Chapters 9 and 10) that the hydrogen bonding motif in the acetanilides,

(8) (and (9)), permits close contact between the aromatic group and the CH_3 group in contrast to the benzanilides, (6) and (7), where lateral interactions along the hydrogen bonding axis require the rings to twist out of the plane of the amide group. This twist of the benzene rings out of the plane of the amide group ($|\tau_2|$ and $|\tau_3|$) reduces the resonance interactions between the rings and the amide group. This accounts for the relatively small influence of the polar substituents on the bonding in the amide function.

The extensive resonance interactions expected in the benzanilides, Ar – C(O) – NH – Ar, are essentially limited by the degree of twist of the benzene rings out of the plane of the amide group. Since this twist is principally as a consequence of intermolecular interactions, which would be absent in solution, through-conjugation is expected to be enhanced in solution. For this reason the substituent has a greater effect on the C = O bond order in solution than in the solid state. Hence the expected influence of the polar substituents on the C = O bond order is not mirrored by differences in the C = O bond length.

The $C - N - C_{Ar}$ bond angle in the amides in Table 11.1 also appears to be related to the degree of twist of the ring out of the plane of the amide group. In cases where τ_2 is large, the $C - N - C_{Ar}$ angle is closer to the expected value of 120°. There is a correlation between the degree of twist of the benzene ring out of the plane of the amide group and the $C - N - C_{Ar}$ angle; this is shown in Figure 11.1. The enlargement of the $C - N - C_{Ar}$ angle is a consequence of repulsions between the carbonyl group and the *ortho* hydrogen atoms on the benzene ring. In amides where co-planarity of the amide group and the benzene

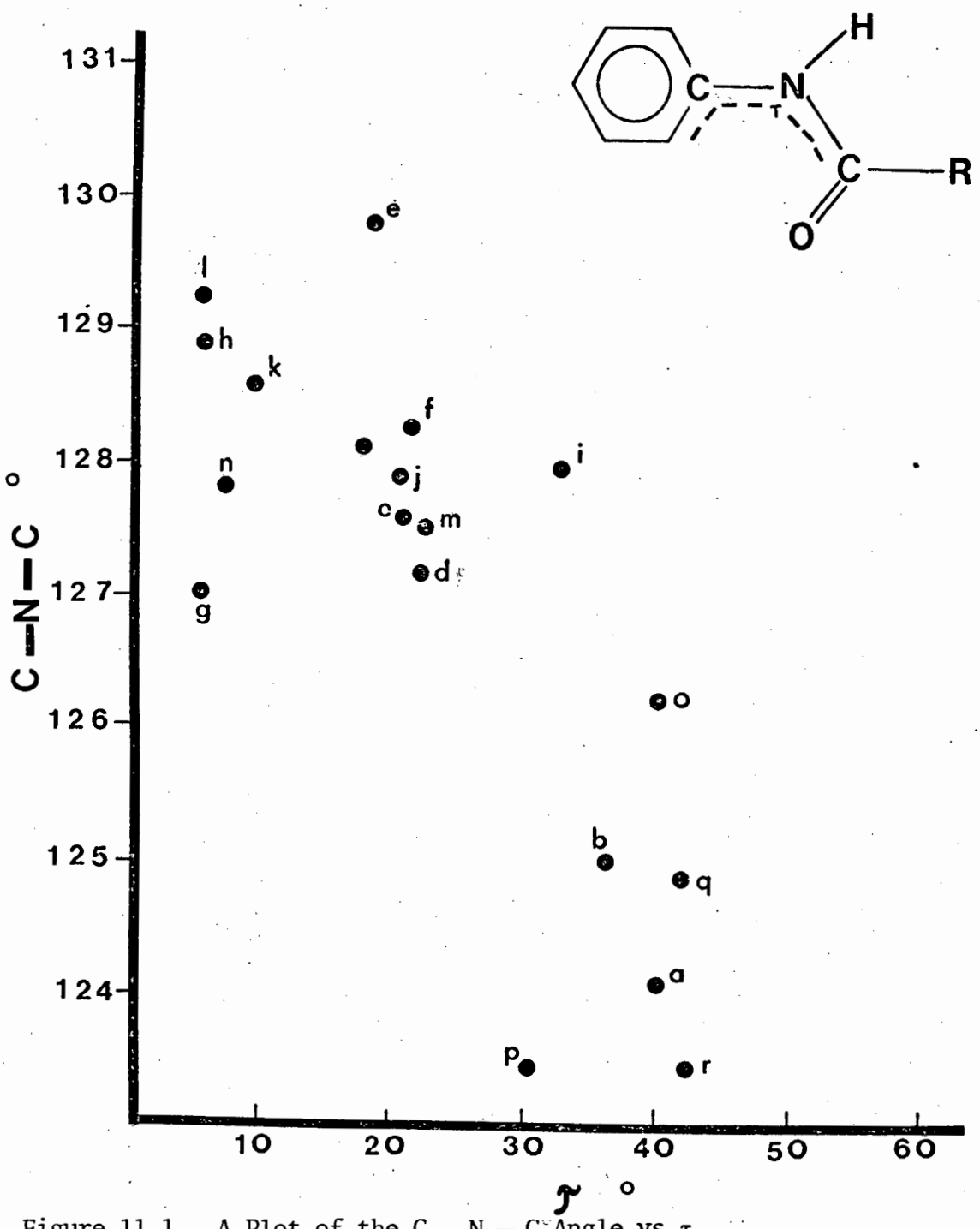
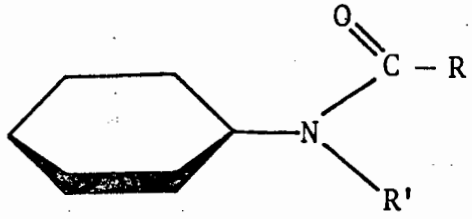


Figure 11.1 A Plot of the C — N — C Angle vs τ

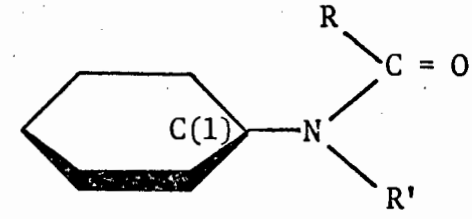
(a) *p*-Methoxybenzanilide (this work), (b) *p*-nitrobenzanilide (this work), (c) acetanilide[45], (d) *p*-methoxyacetanilide (this work), (e) *p*-hydroxyacetanilide (orthorhombic)[75], (f) *p*-hydroxyacetanilide (monoclinic form)[76], (g) *p*-chloroacetanilide[77], (h) *p*-bromoacetanilide[78], (i) *p*-aminoacetanilide[79], (j) *p*-methylacetanilide (monoclinic form)[79], (k) *p*-methylacetanilide (orthorhombic form)[79], (l) 2-methoxyisonitrosoacetanilide[89], (m) 2-ethoxyisonitrosoacetanilide[90], (n) 2-chloroisonitrosoacetanilide[91], (o) chloro-4-dipropylacetanilide[92], (p) chloro-2-trifluoromethyl-5-diethylacetanilide[92], (q) N-2-fluorenylacetamide[74], (r)N-2-napthylacetamide[74].

ring is unattainable, due to other steric factors or intermolecular interactions, interaction between the carbonyl oxygen and C(1) is proposed as an important source of stabilization energy which compensates to some extent for the loss of resonance stabilization on twisting the ring out of the amide plane.[71] The interaction between



endo

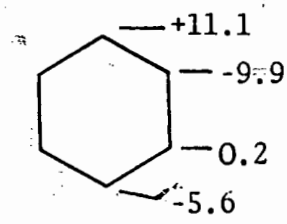
(14)



exo

(15)

O(1) and C(1) in the *endo* form of carboxylic amides (14) is electrostatic in nature and arises because of the δ+ and δ- charge densities on C(1) and O(1) in simple amides (14) and (15)[71]. The electron deficient nature of C(1) in acetanilide is apparent from the ^{13}C n.m.r. shielding parameters (referred to benzene):[93]



The presence of a *para* electron-withdrawing substituent will enhance the positive nature of C(1) and hence favour the *endo* conformation.

Hydrogen Bonding

The hydrogen bonding data for the amides (6)- (8) are summarised in Table 11.2. The carboxylic amides exhibit the translational and 2_1 axis hydrogen bonding motifs[62,63]. The hydrogen bonds in the benzanilides (6) and (7) are relatively weak; the N – H --- O distance is longer than the average distance of 2.85 Å in *trans* amides[60]. The structural results for the carboxylic amides discussed in this section clearly demonstrate the significance of intermolecular interactions to the overall molecular structure. Although the hydrogen bonds in these compounds are relatively weak, the geometrical restraints required to form the hydrogen bond dominate and enforce in some cases an unfavourable conformation on the molecule. Direct comparisons between the properties of the compounds in solution and the solid state properties of these compounds cannot be made out as these hydrogen bonding effects are reduced or absent in solution.

11.3 Phosphoric Amides

The structural data for the phosphoric amides (1) - (5) are listed in Table 11.3. In addition the data for the insecticide crufomate (16)[40], 5,5-dimethyl-2-oxo-2-aminobenzo-1,3,2-dioxophosphorinane (phosphorinane (17))[41] and N-benzoyldimethylphosphoramidate (N-benzoyl, (18))[94] are also included.

Since the P – N distance in the dipolar molecule of phosphoramidic acid $H_3N^+ - PO_3H^-$ (pure single P – N bond) is approximately 1.78 Å[43,95]). Table 11.2 indicates a significant increase in the P – N bond order in

Table 11.2 Hydrogen bond data for the carboxylic amides

Structure	d(N---O) Å	L(N-H---O) (°)	Hydrogen bonding mode	Conformation of the amide group
p-NO_2(benzanilide) (7)	3.112(5)	151.2(9)	translation axis of 5.33 Å	*trans*
acetanilide[45] (10)	2.943(3)	-	2_1 axis of 9.48 Å	*trans*
N-methyl(benzanilide)[62] (9)	2.93 (1)	-	2_1 axis of 9.61 Å	*trans*
p-OCH_3(benzanilide) (6)	3.105(7)	169.3(9)	translation axis of 5.25 Å	*trans*
p-OCH_3(acetanilide) (8)	2.875(5)	177.1(9)	2_1 axis of 9.22 Å	*trans*

Table 11.3 Structural Parameters in the Phosphoramidates

Structure[a]		d(P=O)	d(P-N)	d(N-C_{ar})	τ_1[b]	τ_2[c]	$\angle$(P-N-C_{ar})	α_X[d]	α_{NH}[e]	$(\alpha_X)_s$[f]	$(\alpha_{NH})_s$[g]	ν(P=O)[35] cm^{-1}
p-OMe	(1)	1.462(2) 1.466(2)	1.626(2) 1.630(3)	1.419(4) 1.413(4)	-170.6(3) 176.6(3)	-42.2(3) - 1.9(3)	126.6(2) 130.4(2)	119.3(3) 118.7(3)	118.1(3) 118.3(3)	119.9(1)	119.2(2)	1224
H	(2)	1.458(3) 1.453(4)	1.623(4) 1.604(4)	1.427(6) 1.415(6)	-175.8(5) -177.3(5)	- 7.9(5) - 8.7(5)	130.5(3) 130.7(3)	119.6(5) 118.4(5)	119.7(5) 119.5(5)	-	119.2(2)	1229
p-NO_2	(3)	1.462(5)	1.643(7)	1.406(9)	-178.4(9)	- 0.8(9)	128.9(6)	124.8(10)	120.3(9)	122.1(1)	119.2(2)	1231.5
o-Et	(4)	1.458(3) 1.469(3) 1.456(5)	1.631(4) 1.622(4) 1.629(6)	1.419(5) 1.427(5) 1.425(9)	-175.4(5) -171.2(5) -167.5(9)	- 8.9(5) -27.9(5) -20.7(9)	128.6(3) 127.7(3) 129.2(6)	118.2(4) 119.6(4) 118.1(9)	119.6(4) 119.0(4) 120.9(8)	-	119.2(2)	1242
2,6-diMe	(5)	1.448(4)	1.620(4)	1.436(5)	-29.9(4)	82.9(4)	124.9(3)	119.2(4) 118.1(4)	121.2(4)	117.5(3)	119.2(2)	1237
Crufomate[40]	(16)	1.459(4)	1.611(5)	1.456(7)	-179.4[h]	-	123.4(4)	-	-	-	-	-
Phosphorinane[41]	(17)	1.476(10)	1.649(10)	1.424(9)	33.6[h]	24.3[h]	123.2(4)	-	117.8(9)	-	119.2(2)	-
N-benzoyl[94]	(18)	1.461(4)	1.667(5)	1.393(7) 1.473(8)	179.4(6)[h] 178.0(6)	17.3(6)[h] 16.2(16)	124.7(4)	118.1(6)	-	119.4(2)	-	-

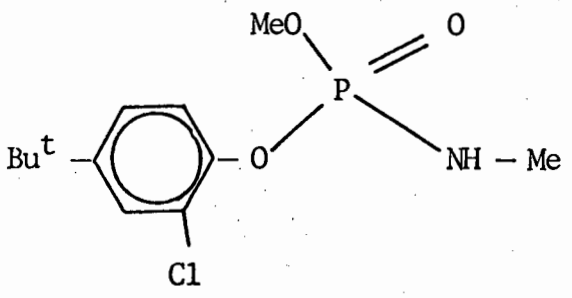
a All bond lengths in Å and angles in degrees

b τ_1 is a measure of the planarity of the phosphoramidate group, i.e. = O(1) – P(1) – N(1) – C(1) torsion angle

c τ_2 = P(1) – N(1) – C(1) – C(2) or P(1) – N(1) – C(1) – C(6) torsion angle

d-g Refer to footnotes to Table 11.1

h These values are not strictly comparable with the phosphoramides (1) - (5) because of the different groups attached to the phosphoramidate group.



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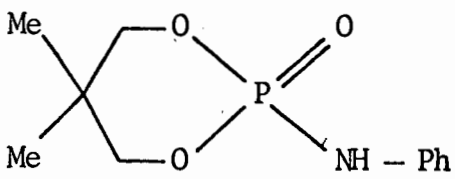

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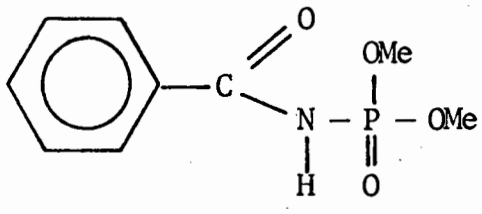
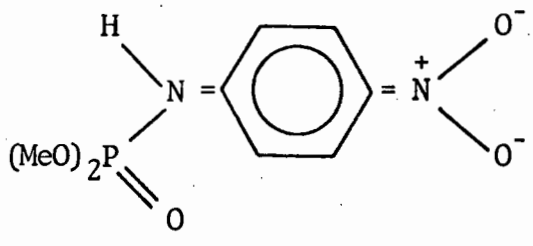


(18)

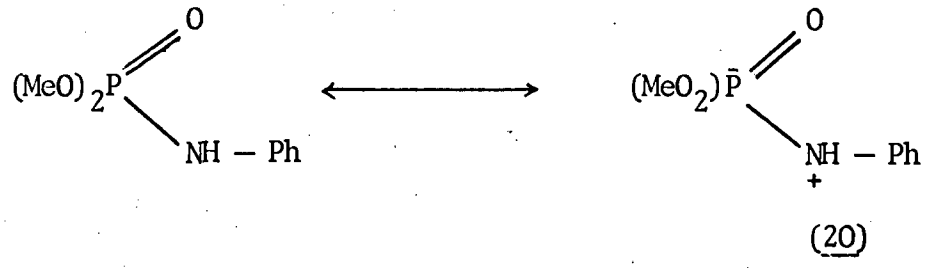
most of the amides studied. This effect is stronger for the N-alkyl derivative (16), since in this compound the nitrogen lone pair electrons are fully available for interaction with the phosphoryl centre. For the substituted phosphoramidates (1) and (3) the ring substitution results in the shortening of the $N - C_{Ar}$ bonds. In the *p*-nitro compound (3) the contribution of the dipolar resonance structure (19) is responsible for the shortest (1.406(9) Å) $N - C_{Ar}$ distance.



(19)

The $N - C_{Ar}$ bonds in the phosphoramidates (1) and (3) are shorter than the corresponding bonds in their carboxylic analogues, (6), (7) and (8). This suggests that the phosphoryl group competes with respect to the nitrogen non-bonding electrons less effectively than the carbonyl group. This is in agreement with the conclusion reached earlier[28] on the basis of the ^{13}C n.m.r. spectroscopy of the N-acylated and phosphorylated aniline derivatives. It must be pointed out however that in the phosphoramidates (1) and (3) the benzene ring is only slightly twisted out of the plane of the amide group, a favourable situation for interaction with the nitrogen non-bonded electrons. In contrast in the carboxylic analogues (6) and (7) the twist of these two groups limits interaction. It is significant however that the value of the $N - C_{Ar}$ bond length in acetanilide ($\tau_2 = 20.0(2)°$) is 1.413(3) Å compared to the average value 1.416(6) Å in the related phosphoramidate (2) ($\tau_2 \simeq 8°$). In the alkyl substituted phosphoramidates (4) and (5) the $N - C_{Ar}$ bond length is lengthened over the unsubstituted analogue (2). In the case of the 2,6-dimethyl compound the long $N - C_{Ar}$ bond length of 1.436(5) Å is a consequence of the minimal interaction between the amide group and the ring ($\tau_2 \sim 83°$).

A feature of the phosphoramidates listed in Table 11.3 is the small difference in the value of the phosphoryl bond length. It can be interpreted in terms of the constant P = O bond order, regardless of the degree of the P_π - d_π back donation effect of the amide nitrogen atom. The relatively constant value of the P = O distance suggests that the resonance contribution of the resonance structure (20), involving an hexavalent phosphorus atom may be considered[96]. The resonance structure (20) accounts for the reluctance of the nitrogen atom to transfer electron density to the phosphoryl group.

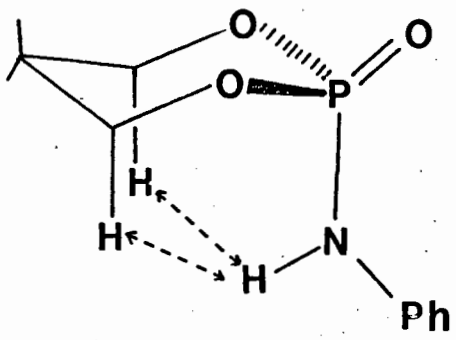


(20)

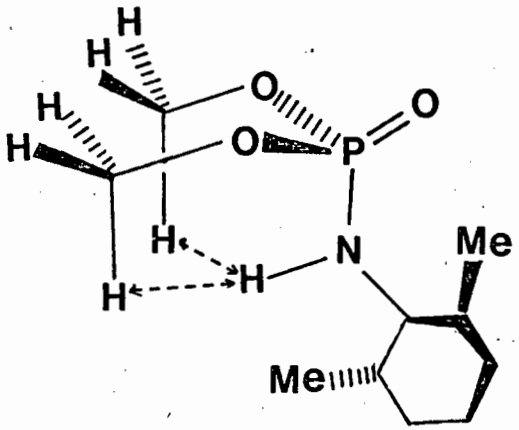
The difference between the resonance interaction between the phosphoryl group and the carbonyl group is apparent in the data for the mixed imide compound (18)[94]. The P – N bond distance of 1.667(5) Å is the largest in Table 11.3 and indicates that resonance interaction between the nitrogen atom and the carbonyl group dominates any interaction between the nitrogen atom and the phosphoryl group.

The molecular parameters of the phosphoramidate group are sensitive to steric requirements. When compared with the non-cyclic analogue (2), the dioxaphosphorinane structure (17) is characterised by relatively long P = O and P – N bonds and non-planarity of the phosphoramidate group (τ_1). This is the result of a tendency by the phosphoryl oxygen and the NHPh group to avoid destabilizing interactions with the 4,6 hydrogen and 5,5-methyl groups of the six-membered ring. These interactions (21) force this compound to adopt the *trans* orientation of the phosphoramidate groups, while most of the other compounds in Table 11.3 exhibit the *cis* orientation. By analogy the 2,6-dimethyl compound (5) also exhibits the *trans* orientation in order to limit interactions between the 2,6-methyl groups and the phosphate ester groups. The steric interactions in this compound result in an angle of almost 90° (τ_2 = 82.9°) between the benzene ring and phosphoramidate group. This twist effectively illiminates any ring interactions with the phosphoramidate group and is seen as a lengthening of the C_{Ar} – N

bond length compared to the unsubstituted compound (2).



(21)



(5)

The lengthening of the $C_{Ar} - N$ bond length may also be the result of the steric repulsions. For example, in the structure of the methyl phenyl phosphoramidate of ethyl L-phenylalaninate, the substitution of the nitrogen atom by the bulky group results in unfavourable interactions between this group and the $(RO)_2P(O)$ moiety. In this compound the distance between the nitrogen atom and the bulky phenylalaninate ester group is increased to 1.49 Å[97,98].

In the phosphoramidates in Table 11.3 the $P - N - C_{Ar}$ angle is enlarged over 120° to reduce repulsions between the phosphoryl group and the *ortho* hydrogen atoms. Figure 11.2 shows a plot of the torsion angle $|\tau_2|$ against the $P - N - C_{Ar}$ angle and the planarity of the amide group $|\tau_1'|$. The $P - N - C_{Ar}$ vs. $|\tau_2|$ (i.e. twist of the benzene ring) plot shows a similar relationship to that found in the carboxylic amides. Although one would have preferred more data points in these plots there is a correlation between the distortion of the amide group and the phosphoramidate group and benzene ring co- planarity. These plots show that in the *p*-substituted analogues the amide group

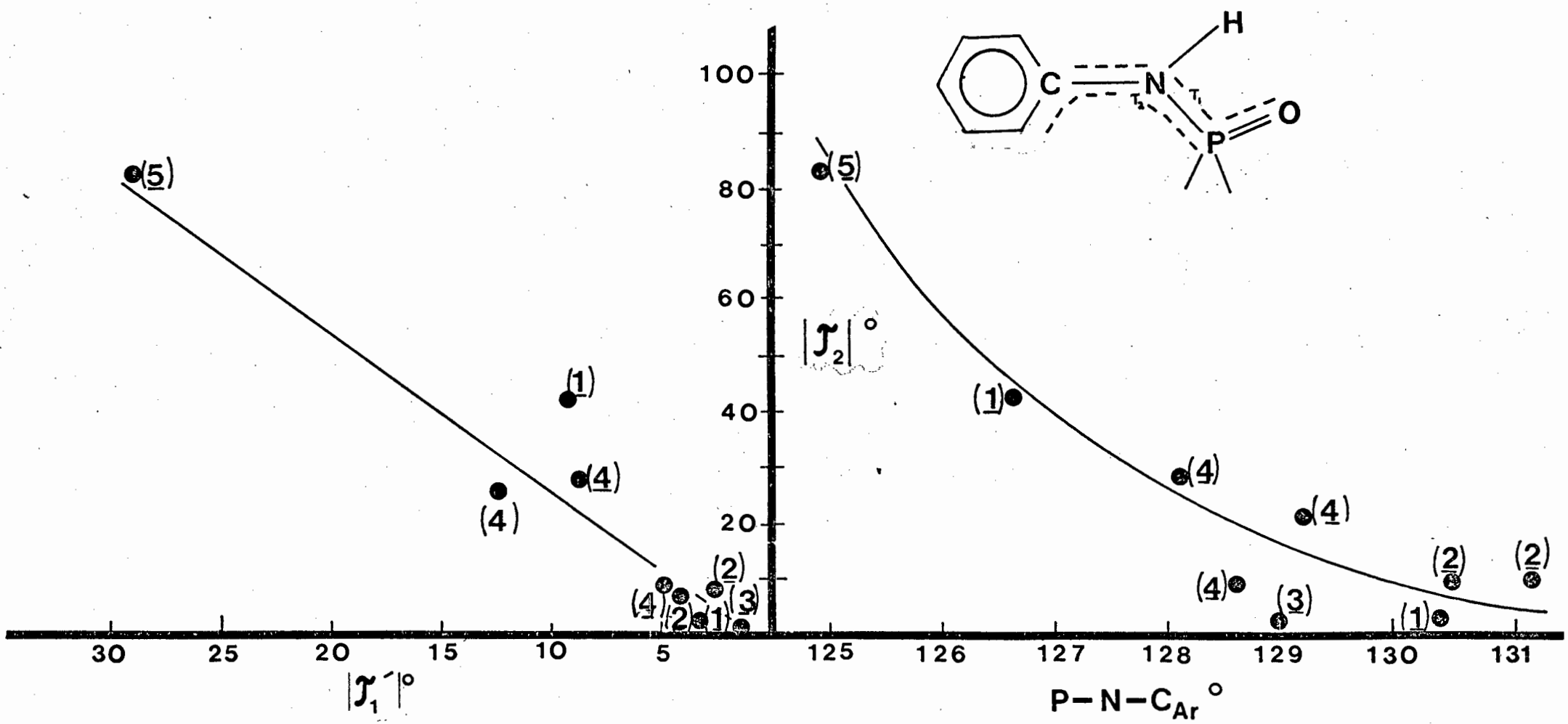


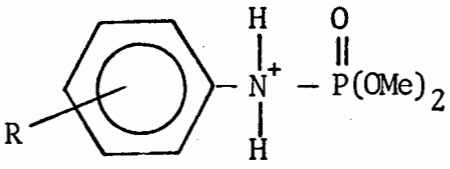
Figure 11.2 A plot of the planarity of the amide group τ_1' vs. the twist of the benzene ring with respect to the amide group $|\tau_2|$ and a plot of the P-N-C_{Ar} angle vs. $|\tau_2|$ for the phosphoric amides
NOTE: $\tau_1' = 180° - |\tau_1|$ if $|\tau_1| > 90° = |\tau_1|$ if $|\tau_1 < 90°$

and the ring are coplanar and distortion of the phosphoramidate from planarity is small. On the other hand the disruption of this situation by the *o*-alkyl substituents in compounds (4) and (5) is apparent.

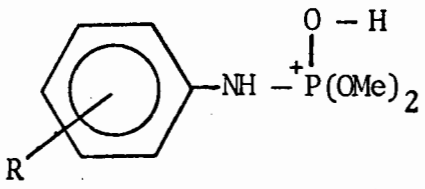
The phosphoramidates (2), (4) and (5) have been the subject of a study of the acid catalysed hydrolysis reaction of these compounds by Modro and Rijkmans:[38]

$$(MeO)_2P(O) - NH - Ar + H_2O + H^+ \longrightarrow (MeO)_2PO_2H + ArNH_3^+$$

In this study the rate of P – N bond cleavage was measured. The findings of this study have shown that the reactive species in this reaction is the N-protonated form (I) rather than the O-protonated form (II) in contrast to carboxylic amides where the reverse is the case[99-101].

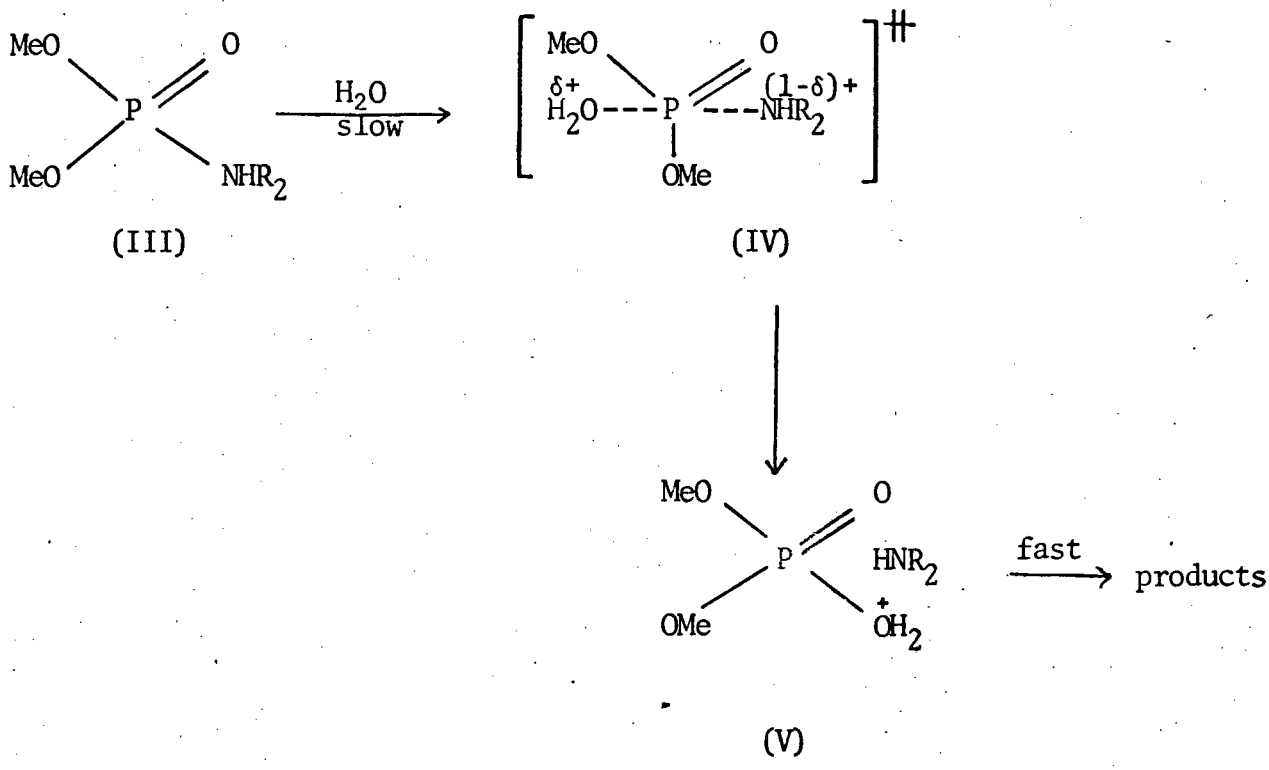


(I)



(II)

The proposed mechanism for this reaction is the so-called A-2 type mechanism where direct substitution of water involving a rate-determining one-step process occurs in which the P – N bond is broken at the same time that the water attacks the phosphorus atom.[26,102] In this reaction it was found that the position of the substituent on the benzene ring had a marked influence on the rate of the reaction. In particular for the *ortho* substituted phosphoramidates there was a sharp change in the reactivity-basicity relationship. It is proposed that the protonated species (III) in *ortho* substituted analogues will



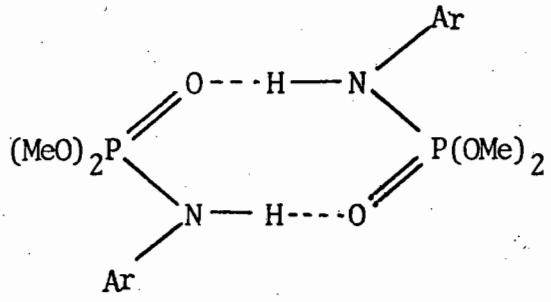
not be stabilized by solvation to as great an extent as the substrates with *meta* or *para* substituents because of crowding in (III), and consequently the reactivity of these phosphoramidates in acidic media will be more sensitive to changes in the basicity of the corresponding anilines. The structural results for the *o*-alkyl substituted phosphoramidates, (5) and (6), support this view. The marked effect of crowding in these compounds is apparent in the severe distortion of the geometry of the phosphoramidate group in comparison with *p*-substituted compounds.

The strong dependence of the hydrolytic reactivity of the phosphoramidates on the hydrating properties of the reaction medium which was observed supports the postulated N-protonated form of the substrate in the hydrolysis scheme. The O-protonated form (II) would be characterized by a significant charge-delocalization. The structural results

(viz. P = O bond length) reveal that there is little delocalization in the phosphoryl group and support this view.

Hydrogen Bonding in Phosphoramidates

The hydrogen bond data for the phosphoramidates (1) to (5) as well as those for the compounds (16) to (18) are listed in Table 11.4. The structural data indicate the preference of the phosphoramidates (1) to (5) for the dimeric hydrogen bonding mode (22). Compounds (2) and (5) are exceptions and exhibit a polymeric hydrogen mode.



(22)

The infrared study (refer to chapter 3) showed that the hydrogen bonding effects persist even in dilute solution in the phosphoramidates (1)-(4) while the phosphoramidate (5) exhibited a marked difference in the properties in the solid state and solution. The reason for this is now clear since the polymeric arrangement in (5) is sensitive to the intervention of solvent molecules. Compound (4), although not as sensitive to solvent effects as (5) also showed enhanced solvent effects on $\nu(P = O)$. The dimeric hydrogen-bonded species in the *o*-ethyl compound (4) is highly crowded and relief from

Table 11.4 Hydrogen bonding data for the phosphoric amides

Structure		d(N---O) Å	∠(N–H---O) (°)	Hydrogen bonding mode	Conformation of the amide group
p-OMe	(1)	2.877(4) 2.886(4)	176.1(3) 168.8(3)	Independent cyclic dimer	*cis*
H	(2)	2.880(4) 2.861(4)	171.1(9) 174.6(9)	Two centrosymmetrically related cyclic dimers	*cis*
p-NO_2	(3)	2.83 (1)	164.4(9)	Glide of 7.42 Å	*cis*
o-ethyl	(4)	2.889(4) 2.915(4)	156.6(6) 164.3(6)	Independent cyclic dimer	*cis*
		2.890(6)	159.8(6)	Centrosymmetrically related cyclic dimer	*cis*
2,6-dimethyl	(5)	2.871(5)	168.5(3)	Glide of 10.28 Å	*trans*
Phosphorusaine[41]	(17)	2.80 (1)	unavailable	2_1 axis of 9.71 Å	*trans*
N-benzoyl[94]	(18)	2.78 (1)	unavailable	centrosymmetric dimer	*cis*

this crowding may be achieved by the formation of hydrogen bonds with the smaller solvent molecules. Although the *p*-nitro compound has a polymeric hydrogen bonding mode the molecule exhibits the *cis*-orientation of the phosphoramidate group and presumably dimerization takes place in solution.

Since the phosphoramidates (1), (2), (3) differ only by a substituent at the para position of the N-aryl group, the variations in the hydrogen bonding can be considered as a direct function of the polar effects of these substituents. The electronic effects of ring substituents should be expected to modify the acidity of the N – H group, thus to vary the strength of the corresponding hydrogen bond. For these three phosphoramidates the hydrogen bonding (measured as the intermolecular N --- O distance) gives a fairly linear correlation with the *pKa* values of the corresponding anilinium ions $Ar - NH_3^+$ (Figure 11.3)[103]. Although the linear free energy relationship represented in Figure 11.3 is an approximate one there seems to be a direct correlation between the acidic properties measured in solution and the intermolecular interactions in the solid state. The plot is a further indication that the electronic effects of the N-substituent are poorly transmitted to the phosphoryl group. If it were otherwise, any increase in donating ability of the N – H function should decrease the accepting ability of the P = O group (and vice versa), so that the net N ---- O distance should not vary significantly with ring substitution.

Figure 11.3A is also further support for the proposal of the N-protonated species in the solvolysis of these compounds. This plot shows that the substituent electron-withdrawal or -releasing effect

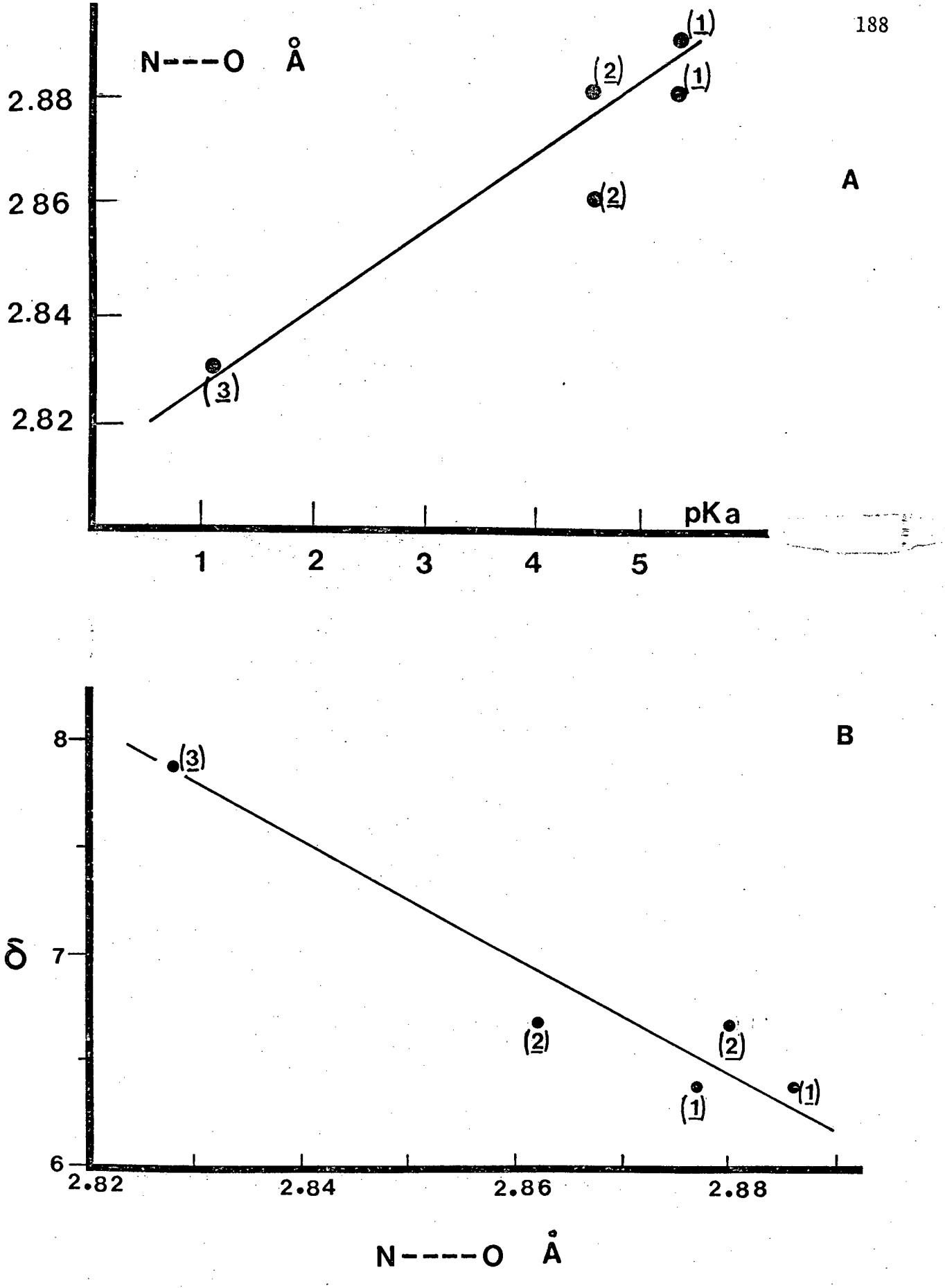


Figure 11.3 A and B. A plot of the N---O hydrogen bond distances vs. the pka values of the corresponding anilinium ions and a plot of the N---O distance against the chemical shift of the amide proton

would directly influence the solvation of the reactive species; however the influence of the substituent on the hydrogen bond is relatively small as the hydrogen bonds are all similar in strength. Further evidence of the sensitivity of the hydrogen bond to the acidity of the amide hydrogen atom is apparent from Figure 11.3B where the hydrogen bond length is correlated with the corresponding ^1H n.m.r. chemical shift (δ)[38] of the amide hydrogen atom.

The hydrogen bonding in the mixed imide compound (18) deserves special mention[94]. The phosphoryl and N – H groups are *syn*-coplanar as opposed to the *anti*-coplanar orientation of the carbonyl and N – H groups. In consequence, the hydrogen bonding involves the formation of a dimer via a pair of N – H --- O = P hydrogen bonds, with no participation of the carbonyl group in the hydrogen bonding. The carbonyl oxygen is located directly beneath the tetrahedral face of the phosphoryl centre containing N, $O^1_{CH_3}$ and $O^2_{CH_3}$. The carbonyl oxygen-phosphorus distance (2.97 Å) is considerably less than the sum of their van der Waals radii (3.30 Å)[104]. This secondary interaction can serve as a model for an "early stage" of nucleophilic substitution at the phosphoryl centre (refer to the next chapter).

Hydrogen Bond Motifs in Phosphoramidates

Cis-phosphoramidates

The predominant motif in the *cis*-phosphoramidates is the dimer (22). The molecules are associated by a pair of P = O ---- H – N hydrogen bonds. The two associated molecules may be related by a centre of symmetry (symmetric dimer) or the two molecules may be crystallographically

independent (asymmetric dimer) resulting in two hydrogen bonds per dimer. The symmetric dimer occurs in compounds (2) and (18) while asymmetric dimers are found in compound (1). Compound (4) crystallizes either as a symmetric dimer or as an asymmetric dimer. In the dimers the phosphoramidate group is generally planar with hydrogen bonds close to linearity. In compound (4) the bulky *o*-ethyl group is still accommodated in a dimeric species at the expense of some distortion of the planarity of the phosphoramidate group and the linearity of the hydrogen bond. Generally the requirements of dimerization are met without significant distortions of the geometry of the molecule, except perhaps the enlargement of the $P - N - C_{Ar}$ angle in the *cis*-orientation of the amide group. This is however as a result of intramolecular repulsions and may even assist dimerization by increasing the distance between the O = P and H – N groups in each molecule.

Compound (3) although a *cis*-phosphoramidate exhibits an intermolecular hydrogen bonding motif. The geometry of this molecule is identical to that of the other *cis*-phosphoramidates which form dimers and so presumably dimerization can occur in solution. The polymeric arrangement in (3) possibly permits the formation of shorter N --- O distances but the N – H --- O angle is slightly bent. Since the phosphoramidate (2) is not a planar molecule (because of the sp^3 hybridized phosphorus atom) the importance of other ring-to-ring interactions similar to those observed in the carboxylic amides are reduced[62,63].

The question arises why the *p*-nitro compound adopts the observed hydrogen bonding motif while there appears to be no impediment to formation of dimers. It has already been mentioned (chapter 5) that the dimer (in *cis* carboxylic amides) achieves a more favourable

intermolecular stack than the intermolecular motif[67]. Energy calculations have also shown that certain carboxylic amides will forego the most favourable hydrogen-bonding motif in order to achieve a better interlayer packing which compensates for the 'poorer' hydrogen bonding[105]. In the *p*-nitro compound there are significant intermolecular interactions other than the hydrogen bonding. These involve interactions between the *p*-nitro groups and the benzene rings of alternate molecules. The presence of these π-π interactions might override the preference of this compound for the dimeric structure.

Trans-phosphoramidates

In compounds (5) and (17) intramolecular repulsions force the *trans* orientation of the phosphoramidate group which results in an intermolecular hydrogen bonding motif. The glide motif in (5) is similar to the glide motif of carboxylic amides[62]. The stack along the hydrogen bonding axis is such that the N-aromatic groups are sharply tilted to the stack axis (Figure 8.2). The sharp twist of the rings to the hydrogen bonding axis permits overlap of each ring with another ring in a ribbon parallel to it. Therefore the twist of the benzene ring out of the plane of the amide group not only reduces intermolecular interactions but also permits favourable overlap in packing of the molecules. It is conceivable that the sharp twist of the rings is partly a consequence of the hydrogen bonding motif (as in the carboxylic amides) and would be reduced in solution.

The Hydrogen Bond Length in Phosphoramidates

In a recent study the N --- O distances in carboxylic amides were found to be between 2.60 and 3.15 Å [106]. The NH --- O bond lengths for both *cis* and *trans* carboxylic amides have been estimated at 2.85 Å[62]. The average value for this bond in the phosphoramidates in Table 11.4 is 2.86 Å.

The Symmetry of the Phosphorus Group in the Phosphoramidates (1) - (5)

In previous chapters the geometry of the phosphorus group was referred to as 'distorted tetrahedral' or perhaps 'approximately tetrahedral'. Mathematically this is meaningless since a molecule either possesses a certain symmetry or it does not - the concept of approximate symmetry is meaningless. Nevertheless the concept of approximate symmetry does give an idea of the deviation of a particular group or molecule from a well defined symmetry. There are various ways of describing distorted tetrahedral molecules [107,108] and perhaps the simplest is Baur's method[109]:

A measure of the degree of distortion can be obtained by calculating for every tetrahedron three distortion indices (DI):

$$DI(PX) = \left(\sum_{i=1}^{4} |PX_i - PX_m|\right) / 4PX_m$$

$$DI(XPX) = \left(\sum_{i=1}^{6} |XPX_i - XPX_m|\right) / 6XPX_m$$

$$DI(XX) = \left(\sum_{i=1}^{6} |XX_i - XX_m|\right) / 6XX_m,$$

where PX_i stands for the individual distances from the tetrahedral phosphorus atom to the ligand (O or N in this case), XPX_i for the individual angles X – P – X, XX_i for the individual lengths of the tetrahedral edges and m signifies the mean value for the polyhedron. There are five possible combinations of distortion indices and these are listed below in Table 11.5 (from reference 108).

Table 11.5 Possible Combinations of Distortion Indices which Define Different Distortion Cases

Case	A	B	C	D	E
DI(PX)	=0	≠0	≠0	=0	≠0
DI(XPX)	=0	≠0	=0	≠0	≠0
DI(XX)	=0	=0	≠0	≠0	≠0

Cases A and E are the extreme cases and A represents an ideal tetrahedron. The distortion indices for the phosphoramidates (1) - (5) and (18) are listed in Table 11.6 and strictly speaking all the phosphorus tetrahedra belong to case E (not surprising since the phosphorus atom lies at a general position). Despite this, inspection of the values in Table 11.6 shows that DI(XPX) is almost double DI(XX) and is always larger than DI(PX). Therefore the phosphoramidates tend towards case B in Table 11.5. Case B illustrates a tetrahedral group in which the ligand atoms are equidistant from each other and outline a regular tetrahedron, and the central phosphorus is moved away from the centroid of the tetrahedron (refer to Figure 11.4). In this case one P – X length is longer than the other three. In the phosphoramidates above

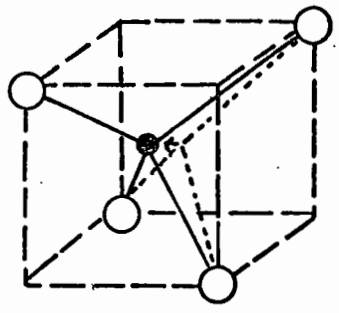


Figure 11.4 Distortion of the symmetry of an ideal tetrahedron corresponding to Case B in Table 11.5. The central phosphorus is moved away from the centroid of the tetrahedron

the P – N bond is longer than the other three bonds.

Table 11.6 Distortion Indices for the Phosphorus Group in the Phosphoramidates (1) - (5) and (18).

Compound	DI(PX)	DI(XPX)	DI(XX)
(1)	0.031	0.044	0.017
(2)	0.030	0.047	0.029
(3)	0.034	0.045	0.020
(4)	0.025	0.044	0.024
(5)	0.036	0.057	0.028
(18)	0.036	0.042	0.033

It must be stressed that the above treatment was originally derived for PO_4^- tetrahedra. This treatment does not strictly apply to the PO_3N group in the phosphoramidates since, while the ideal symmetry of a PO_4^- group is a tetrahedron, the ideal symmetry of a PO_3N group cannot be a tetrahedron (C_{3v}). This treatment does however give one some idea of the shape of the PO_3N group in the phosphoramidates.

The distortion of the phosphoramidates from an average PO_3N structure can be calculated. The parameters for this average structure are determined from the parameters of the structures in Table 11.6. The parameters for this structure are listed in Table 11.7.

Table 11.7 Structural Parameters for the Average PO_3N Structures (C_{3v} Symmetry)

$P = O_1$:	1.459 Å	$O_1 - P - N$:	110.7°
$P - O_{2,3}$:	1.566 Å	$O_1 - P - O_{2,3}$:	115.4°
$P - N$:	1.630 Å	$O_2 - P - O_3$:	98.6°
		$O_{2,3} - P - N$:	108.0°

In the calculation of distortion indices with respect to this average structure the deviations of individual parameters from the mean values listed above are calculated, instead of the mean values of parameters for individual PO_3N groups as before. This takes into account the different mean values of the P = O, P – O, P – N, O = P – O, O – P – O, O = P – N, O – P – N, O --- O and O --- N parameters. The values of $4PX_m$, $6XPX_m$ and $6XX_m$ are obtained from the average structure. The values of the distortion indices calculated in this way are listed in Table 11.8.

Table 11.8 Distortion Indices of the Phosphoramidates with Respect to the Average PO_3N Structure

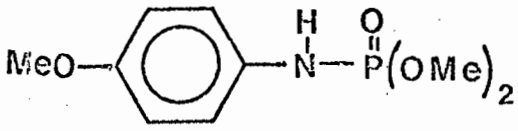
Compound	DI(PX)	DI(XPX)	DI(XX)
(1)	0.006	0.011	0.012
(2)	0.004	0.013	0.011
(3)	0.010	0.017	0.005
(4)	0.003	0.007	0.008
(5)	0.011	0.028	0.017
(18)	0.012	0.010	0.018

Generally the distortion indices are small compared to those in Table 11.6 (and in terms of the values for PO_4^- tetrahedra found by Baur.[109]) Generally therefore the phosphoramidates above exhibit PO_3N groups only slightly distorted from the shape of the average PO_3N group. Compound (5), the 2,6-dimethyl substituted compound shows the largest deviation from the ideal structure.

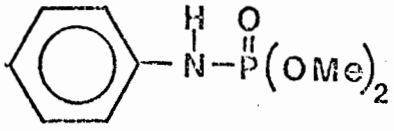
11.4 Conclusions

The structural results for the phosphoramidates, (1) - (5), show that although the substituents interact with the benzene rings in the expected way (determined from the *ipso* angles of the substituents - see Table 11.3) these effects are not transmitted beyond the nitrogen atom to the phosphoryl group. This is the most important difference between the carboxylic and phosphoric amides since it is well established that the amide group in carboxylic amides is extensively conjugated. The phosphoric amides are less subject to the influence of intermolecular interactions which can distort the preferred geometry of the molecules, than the carboxylic amides.

Many of the structural features of the phosphoramidates are unusual and therefore the structural determinations have been a satisfying exercise. There is however scope for more work in this area in order to assess whether the correlations discussed in this work are found in other structures.



(1)



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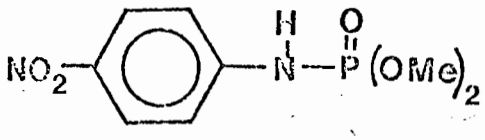

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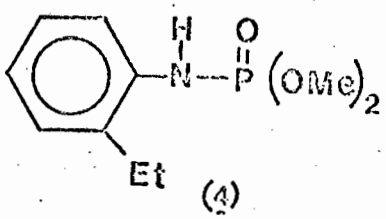

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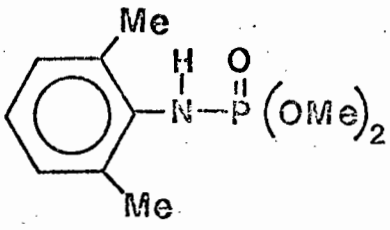

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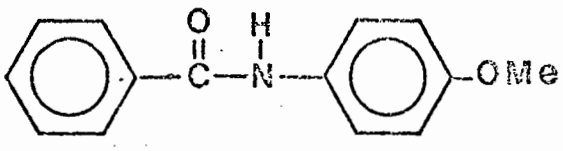

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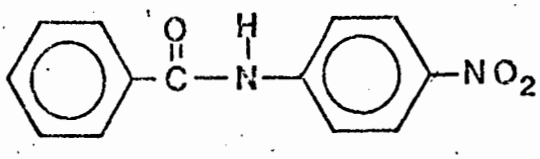

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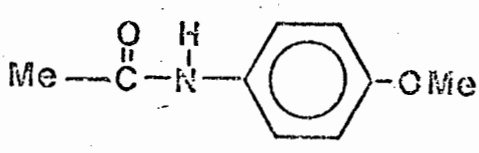

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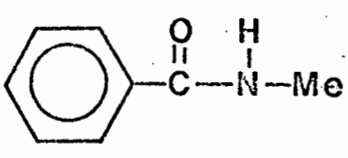

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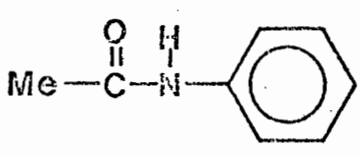

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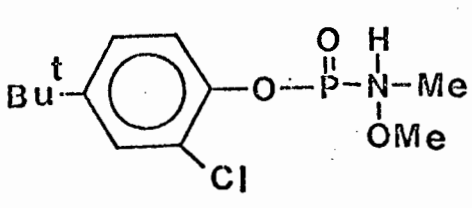

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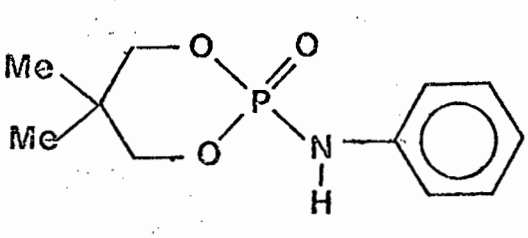

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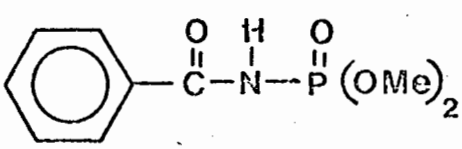

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PART A : CHAPTER TWELVE

12. THE STRUCTURAL CORRELATION PRINCIPLE

12.1 Introduction

The course of a chemical reaction is described by an energy landscape or hypersurface (Born-Oppenheimer surface). Paths joining energy minima on this hypersurface, via its passes, are called reaction pathways, and every reacting system seeks out such a low energy pathway. The simplest system for which such a pathway has been described is the linear H_3 system - i.e. the $H_2 + H \rightarrow H + H_2$ exchange process. The potential energy surface for this system is shown in Figure 12.1[110].

In a 1968 review of donor-acceptor interactions[111] Bent suggested that "certain kinds of attractive intermolecular interactions may be viewed as incipient valence shell expansions and often as the first stage of bimolecular nucleophilic displacement reactions". In particular the correlation between the I --- I distances observed in the I_3^- anion in crystal structures revealed the curve shown in Figure 12.2.[2] Bent suggested that "the hyperloid curve may be presumed to show, approximately, the changes that occur in the distances between nearest neighbours in the linear exchange reaction $I^1 + I^2I^3 = I^1I^2 + I^3$. In addition similar relationships are found in (b) thiathiophthenes and (c) O – H --- O hydrogen bonds.

The resemblance between the curves in Figure 12.2 and the minimum energy path for the H_3 system was obvious. Such similarities between these two curves held out the hope that the structural changes that occur along a reaction pathway could be mapped out since the individual

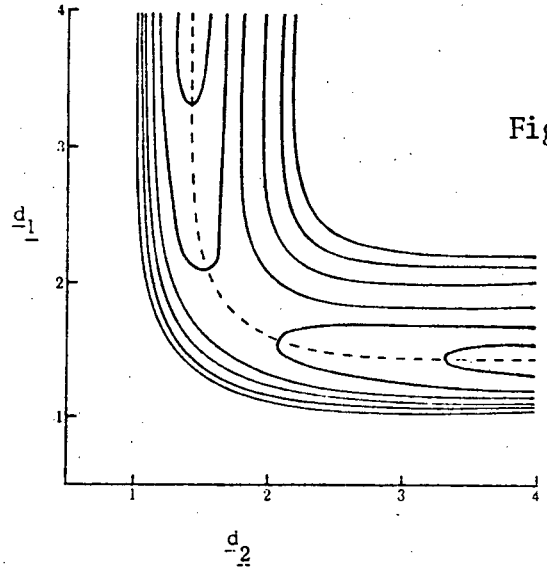
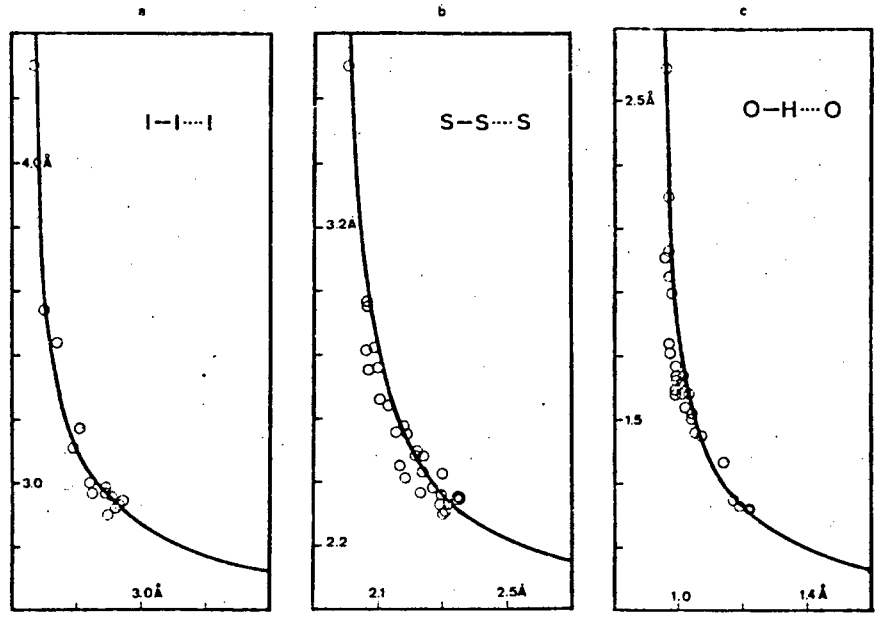


Figure 12.1 (from ref. 9)

Potential energy surface for linear H_3 system, drawn from data calculated by Liu [*J. Chem. Phys.* 58, 1925 (1973)]. Distances are in bohr units (1 bohr = 0.529 Å), and contour lines are drawn at energy intervals of 0.01 hartrees (1 hartree = 27.21 eV).



Correlation plots of interatomic distances in linear triatomic systems. (a) Triiodide anions, (b) thiathiophthenes, and (c) O—H···O hydrogen bonds. From Bürgi, *Angew. Chem., Intern. Ed.* 14, 460 (1975).

Figure 12.2 (from ref. 9)

points on the curves in Figure 12.2 represent different stages along the energy pathway. However one should refrain from referring to these points as transition states as they may correspond to saddle points or to dips in the energy surface.

The correlation curves in Figure 12.2 have a special analytical form derived from Pauling's intuitive model of chemical bonding:[112]

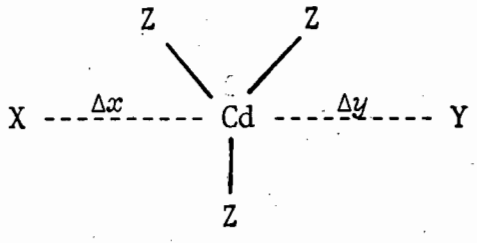
$$d(n) - d(1) = \Delta d = -c\log n \qquad (12.1)$$

The length of a fractional bond d(n) is related to that of a single bond d(1) by this relation. The symbol n is called the bond number and c = 0.60 Å. Pauling defined the bond number, n, as "the number of shared electron pairs involved in the bond".[112] The value of c varies somewhat depending on the nature of the atoms involved. This Pauling expression was applied to the linear triatomics making the assumption that the sum of the two bond numbers equals unity for all related pairs of distances. This assumption implies conservation of bond number along the minimum energy path. This leads to the relationship:

$$10^{-\Delta d_1/c} + 10^{-\Delta d_2/c} = 1 \qquad (12.2)$$

where Δd_1 and Δd_2 are related bond distance increments. The constant, c, may be evaluated from the expression $c = \Delta d^1/\log 2$, where Δd^1 is the bond distance increment in the symmetrical structure ($n = \frac{1}{2}$). Now d(1), the single bond distance to which Δd increments refer, and $\Delta d^1 = d(\frac{1}{2}) - d(1)$ are evaluated to calculate c. Based on the evaluation of these constants the curves in Figure 12.2 were produced from equation 12.1.

This approach has been applied to more complex systems. In particular the structural data pertaining to five coordinated cadmium complexes in various crystal environments were evaluated in terms of the changes occurring in the course of the nucleophilic displacement at tetrahedrally coordinated cadmium[113]. This involved the evaluation of the correlation between the distances x and y brought to a common basis by subtracting the appropriate sums of covalent radii (to obtain Δx, Δy), or the correlation between Δx and Δy and the displacement of the Cd atom from the plane of the three equatorial atoms (sulpher) (I).



(I)

The correlation plot of Δx and Δy shows the same general form as that obtained for the triatomic system and hence an appropriate analytical expression was derived.

It is important to note that the groups X and Y need not belong to the same molecule. If the group Y is bonded to the central atom the X atom (approaching nucleophile) may involve an intramolecular close approach to the central atom or it may belong to another molecule in the crystal structure. It is for this reason that close contacts or secondary interactions have assumed a new significance. By considering the secondary interactions to Sn(IV)[114] and Zn(II)[115] with different coordination numbers analytical curves of the same form as Figure 12.2 have been obtained.

The distortions of the symmetry of a central atom may also be analysed to yield analytical curves. This approach is particularly applicable to tetrahedral molecules and ions which participate in a S_N1 type process where no nucleophilic approach is involved in the initial (i.e. early) stages of a reaction pathway. In such a situation the tetrahedral symmetry of the central atom is distorted and may be described as C_{3v}. The C_{3v} type distortions of a tetrahedron require three parameters for their analysis and are given in Figure 12.3. The analytical curves describing such systems are given in Figures 12.4, 12.5, 12.6 and 12.7 and describe the systems OSO_3, YSO_3, OPO_3, $ClAlCl_3$, YPO_3 and $YSnCl_3$.[116] Particularly important is the fact that these five curves can be reduced to a single analytical curve with reference to a common origin by subtracting a standard value from the observed bond lengths (Figure 12.7). These curves may be described by the functions:

$$\Delta r_2 = -c \log (9 \cos^2\theta) \qquad \Delta r_1 = -c \log (\tfrac{4}{3} - 3 \cos^2\theta) \qquad (12.3)$$

From a least squares fit of the points in Figure 12.7 the value of c is obtained yielding the values below:[116]

Bond	r_o (tetrahedral)	c
Al-Cl	2.125 Å	0.49 Å
Sn-Cl	2.227 Å	0.48 Å
Sn-O	1.472 Å	0.51 Å
P - O	1.534 Å	0.47 Å

Another approach has been to consider the directional preferences of approach of nucleophiles to tetrahedral ions. In an analysis of the

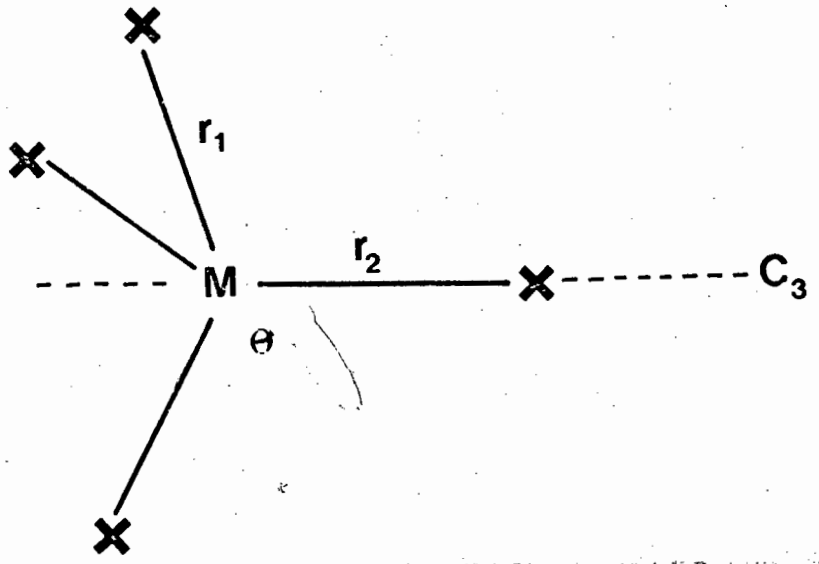


Figure 12.3 The three structural parameters describing a molecule with C_{3v} symmetry

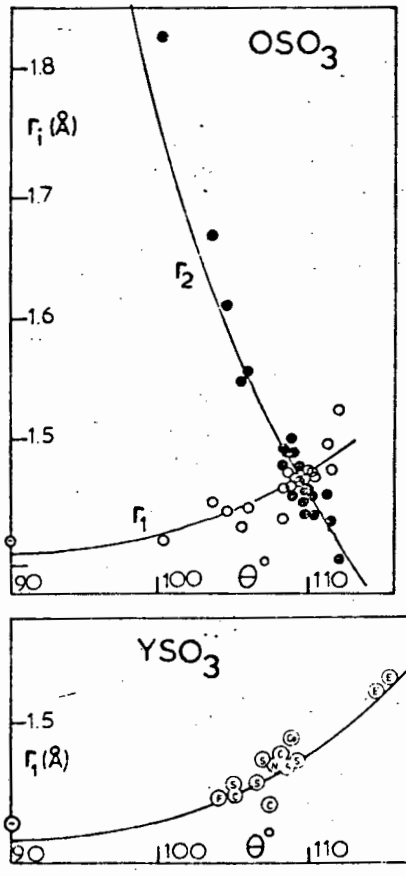


Figure 12.4 (from ref. 9)

Correlation plots for OSO_3 and YSO_3 tetrahedra with approximate C_{3v} symmetry observed in various crystal structures. Black circles show r_2,θ correlations, open ones show r_1,θ. The letters in the open circles define the element Y in YSO_3 (E = electron pair, Θ = planar SO_3). From Murray-Rust, Bürgi, and Dunitz, *J. Am. Chem. Soc.* **97**, 921 (1975).

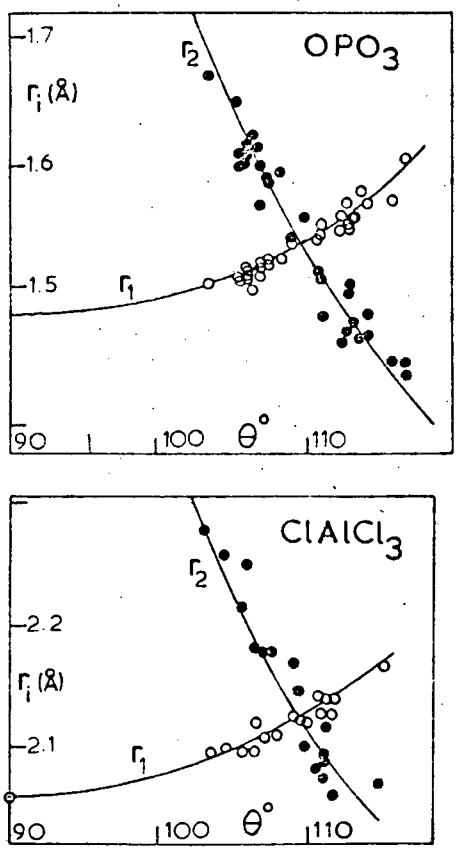


Figure 12.5 (from ref. 9)

Correlation plots for OPO_3 and $ClAlCl_3$ tetrahedra with approximate C_{3v} symmetry. From Murray-Rust, Bürgi, and Dunitz, *J. Am. Chem. Soc.* **97**, 921 (1975).

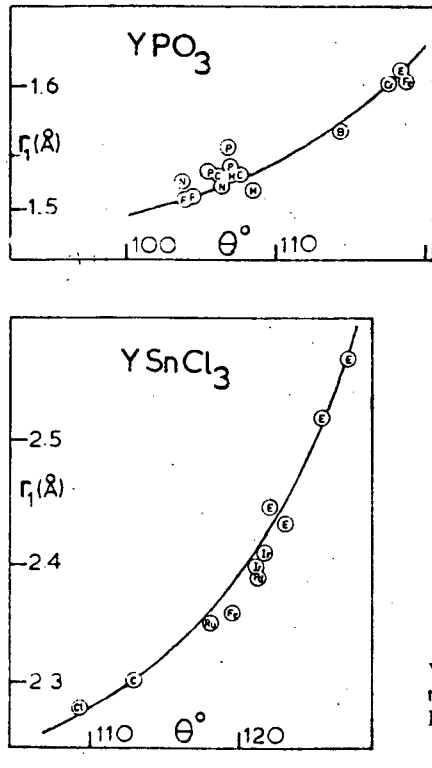


Figure 12.6 (from ref. 9)

Correlation plots for YPO_3 and $YSnCl_3$ tetrahedra. Letters in the circles denote the element Y. From Murray-Rust, Bürgi, and Dunitz, *J. Am. Chem. Soc.* **97**, 921 (1975).

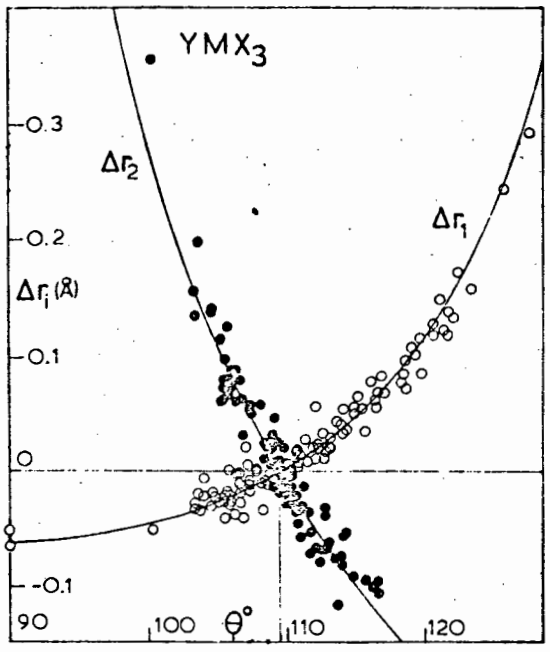


Figure 12.7

Correlation plots for all experimental data shown in Figures 12.4, 12.5 and 12.6 referred to a common origin. The smooth curves are drawn for c = 0.50 Å (from reference 9)

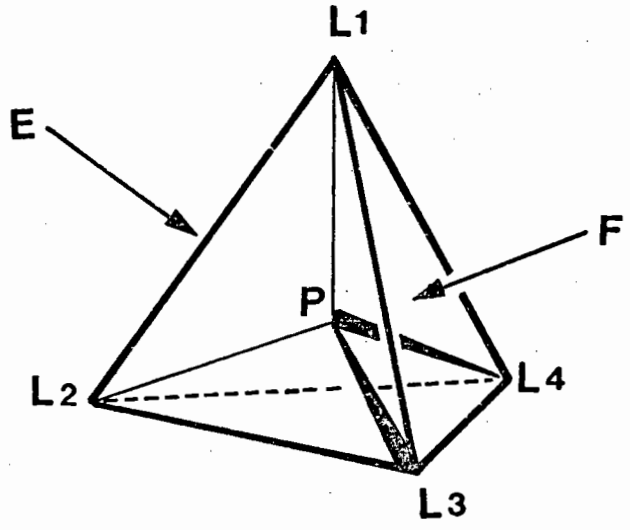


Figure 12.8 The face (F) and edge (E) directions of attack of a nucleophile on a phosphonium ion (PL_4^+)

crystal environments of sulfonium ions[117] Britton and Dunitz showed that short non-bonded contacts (secondary bonds) to sulphur tend to occur along the extensions of the C - S primary bonds. "A single S ---- X^- contact" (X^- = nucleophile) "opposite a primary bond can be regarded as an incipient stage of an addition reaction leading to a sulfurane or a nucleophilic displacement reaction at sulphur." A similar investigation has been made of the approach of a nucleophile to quaternary phosphonium centres[118] (see below).

The correlations between different kinds of parameters discussed above are called the "Structural Correlation Principle"[9]. If the correlations between the various parameters of a fragment can be ordered in some sort of sequence in terms of the gradual deformation of the fragment the resulting sequence may be viewed as a series of frames or "stills" in a cinematic film sequence. Each sample point corresponds to an energy minimum under the perturbation operative in the structure. A word of caution must be considered and to quote Dunitz' authoritative review:[9] "If the smooth curve through the experimental sample points is accepted as a minimum energy path, then by hypothesis the energy must increase along directions normal to this curve. Unfortunately, we do not know how sharply the energy rises normal to the curve, but even worse we know nothing about the energy variation along the curve - i.e. about where the minima and maxima occur ..... To fill out the picture we require a background drawn from other sources. The search for correlations among structural data can be carried out as an exercise in itself, but the patterns that emerge will undoubtedly become more significant when set against the facts of experimental chemistry, their interpretation in mechanistic terms, and the results of quantum mechanical studies".

It is clear therefore that although the 'StructuralCorrelation Principle' appears to lead to minimum energy paths and simple analytical functions they lack theoretical basis. The processes studied must of necessity have a low activation energy and there is disagreement whether the observed structural differences in crystal structures are representative of the differences between isolated species along a reaction pathway. These kinds of questions will only be resolved by further investigations.

12.2 Interactions of Nucleophiles with Quaternary Phosphonium Centres

Archer, Modro and Nassimbeni[118] have reported the preliminary results of the approach of nucleophiles (X^-) to quarternary phosphonium salts. In general, a phosphonium ion can react with a nucleophile (or a base) in a variety of ways, the most important of which are:[119] (i) nucleophilic attack at the positively charged phosphorus atom (formation of a pentavalent intermediate), or (ii) abstraction of the α-proton from one of the substituents (ylide formation). For the reaction pathway (i) two distinct directions of approach are possible.[120] In the so-called "face" attack, the nucleophile meets less steric interference, but once the new bond is formed, group X is in a more congested (apical) position in the tbp product. The alternative "edge" approach, sterically more hindered, results in a less crowded (equatorial) location of the incoming nucleophile (refer to Figure 12.8).

The aims of this part of the research project are to extend and complete the study on the application of the "Structural Correlation Principle" to the quarternary phosphonium ions. This will be described in the next chapter.

CHAPTER THIRTEEN

13, THE APPROACH OF NUCLEOPHILES TO TETRAHEDRAL PHOSPHONIUM CENTRES

13,1 Introduction

A search of the Cambridge Crystallographic Data Centre (CCDC) file was carried out[121] for structures corresponding to the general formula $L_4P^+ X^-$. The search was undertaken on 30 058 entries and revealed 245 structures of the formula above. Of these 36 were chosen for detailed analysis. The remaining compounds found in the CCDC file were not subjected to a detailed analysis since the LP^+ group was generally a counter ion to a complex organometallic compound. The compounds are identified in the CCDC file by a six letter code and the compounds included in this study are listed in numerical order in Appendix 2.

The compounds included in this study included those previously analysed by Archer, Modro and Nassimbeni[118]. The compounds consisted of 19 compounds with the general formula $R_4P^+X^-$[122-139]; one compound of the form $R_3P^+OR'X^-$[140]; one compound of the form $RP^+(OR')_3X^-$[141]; one compound of the formula $R_2P^+(OR')_2X^-$[140]; two compounds of the formula $R_3P^+NR'X^-$[142,143]; two compounds of formula $R_3P^+OHX^-$[144,145]; one compound of formula $R_3P^+ClX^-$[146]; one compound of formula R_3P^+-$R'N^-$[147]; and two compounds of the general formula R_3P^+-$R'O^-$[148,149]. The above compounds were all included because they all exhibit a group that was considered as a potential leaving group in an S_N2 type reaction. The remaining compounds were all of general formula $R_4P^+X^-$ where the groups, R, were a combination of phenyl, methyl or ethyl groups[150-155]. In all the structures the group X^- was either a simple monoatomic ion like Br^-, Cl^- etc. or a more complex ion like $CdCl_4^{2-}$ etc.

13.2 Statistical Analysis

Secondary Interactions

Following the example of Dunitz[117] and Archer, Modro and Nassimbeni[118] an analysis of the P^+----X^- interactions is to be carried out. These interactions are considered in terms of a nucleophile (X^-) approaching a phosphonium ion (L_4P^+). In order to do this, criteria must be established with regard to what P^+ ---- X^- distances are significant since a large range of P^+----X^- distances occur in these structures. This is a contraversial issue and despite the existence of the kinds of correlation curves relating intermolecular distances discussed in the previous chapter, there is some scepticism as to whether some of these 'secondary bonds' can be considered as 'bonds' at all.[1] Alcock has loosely defined a secondary bond as an interaction causing an interatomic distance lying between the expected covalent and van der Waals distances[156]. Despite this it is considered that some interatomic distances up to 1 Å greater than the sum of the van der Waals radii represent weak secondary interactions. This also depends on the actual values of the van der Waals radii used, since methods of the determination of these radii are still being developed. For this work the values of Bondi[157] will be used since these were deduced in a more sophisticated manner than the well-known values of Pauling.[112] The van der Waals radii used are listed in Table 13.1.

Table 13.1 van der Waals Radii [157]

Element	dv (Å)
C	1.70
N	1.55
P	1.80
O	1.52
F	1.44
Cl	1.75
Br	1.85

Angular Dependence of the Approaching Nucleophile

In Figure 13.1 the interatomic distance P^+ ---- X^- in excess of the van der Waals distance, Δd, is plotted against all four $L - P^+$ ---- X^- angles, where $L - P^+$ ---- X^- are the angles between each ligand on the phosphorus atom and the incoming nucleophile (X^-). The compounds in Figure 13.1 represent all the compounds which have an identifiable leaving group bonded to the phosphorus. The Δd values are obtained from the interatomic distance of the phosphorus and the closest nucleophile. In the case of complex ions like $CdCl_4^{2-}$, the value of Δd would refer to the shortest P^+ ---- Cl distance since $CdCl_4^{2-}$ may be regarded as a precursor of Cl^- and $CdCl_3^{2-}$.

If we consider the two possible ways a nucleophile can approach an ideal

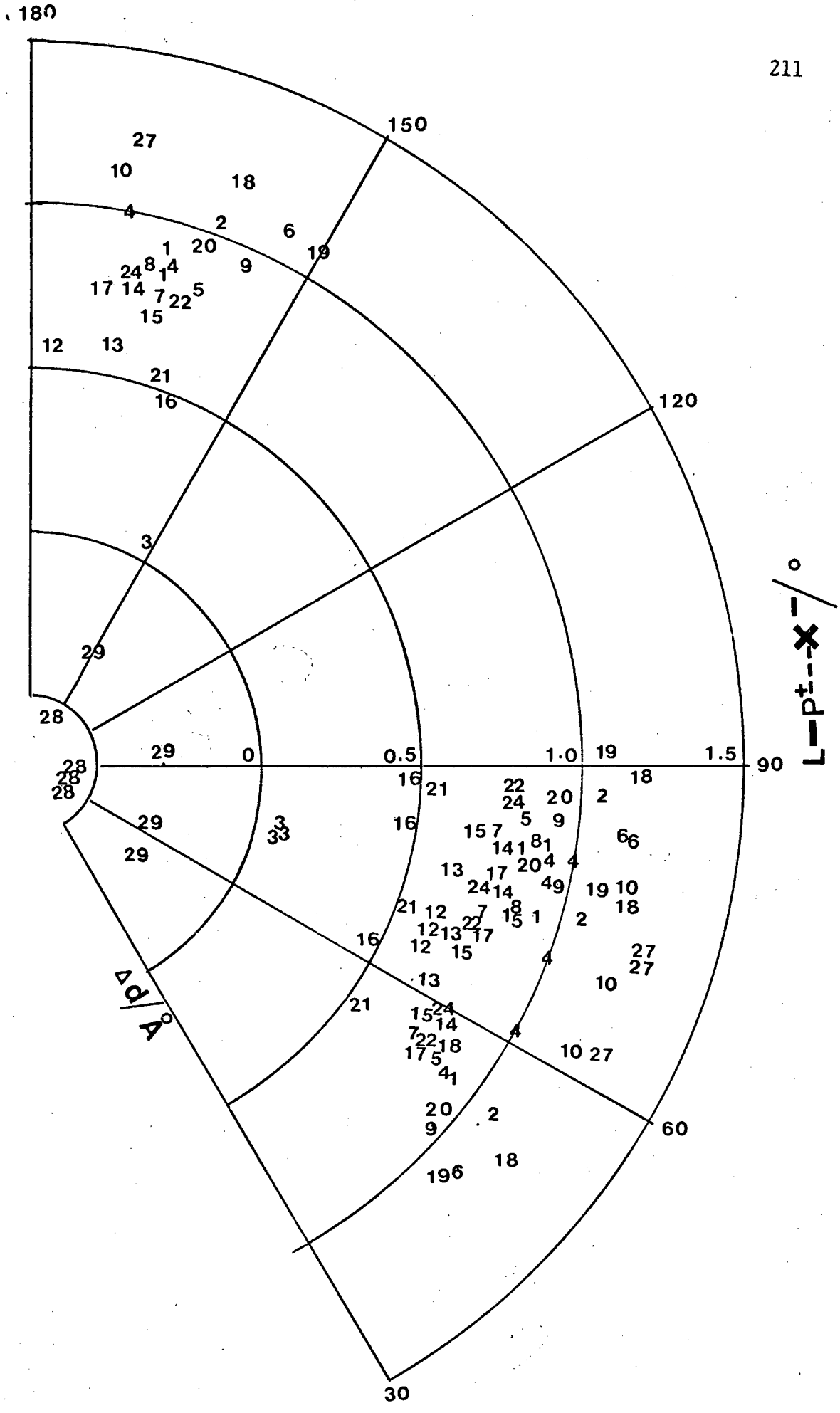


Figure 13.1 A plot of Δd vs. $L - P^{+}\text{----}X^{-}$ Angles

tetrahedron, refer to Figure 12.8, the "face" attack is characterised by $L - P^+ \cdots X^-$ angles of 180° and 70.5°, whereas the "edge" attack is characterised by $L - P \cdots X^-$ angles of 54.7° and 125.3°. In Figure 13.1 the largest $L - P^+ \cdots X^-$ angles lie near 180° and the other angles cluster between 40° and 90°. There is a complete absence of points close to 120° and it is apparent that the $L - P^+ \cdots X^-$ angles indicate a preference for "face" attack.

The directional preferences of the approaching nucleophiles may be represented more clearly in terms of polar coordinates[117] (Figure 13.2). If the phosphorus atom is placed at the origin, the angle θ is simply the angle $L_1 - P^+ - L_i$ ($i = 2,3,4$) or $L - P^+ \cdots X^-$ and ϕ is the angle $L_2 - P^+ - L_j$ ($j = 3,4$) or $L_2 - P^+ \cdots X^-$, measured anticlockwise, projected onto the plane perpendicular to the vector $L_1 - P^+$. Arbitrarily, L_1 (the leaving group) is placed at the "north pole" ($\theta = 0$), and L_2 at the position corresponding to $\phi = 0$. For each ligand (L_2, L_3 and L_4), θ has a value near the tetrahedral angle, 109.5°, and ideally L_3 and L_4 have values of $\phi = 120°$ and 240° respectively.

For each compound the values of θ and ϕ have been calculated. The method of calculation is given in Appendix 3. The relationship between θ and ϕ may be represented by a Mercator Projection (a representation of the surface of a sphere commonly used by cartographers). A plot of these two parameters, θ and ϕ, is shown in Figure 13.3 for all the structures analysed in Figure 13.1. The directions of the ligands attached to the phosphorus atoms (L_{1-4}) appear as close groupings around the points $\theta = 110.7°$; $\phi = 0°$, 120°, 240° (tetrahedral angles). The points representing $P^+ \cdots X^-$ directions are distributed more loosely around the values $\theta = 60°$; $\phi = 60°$, 180° and 300°. By considering

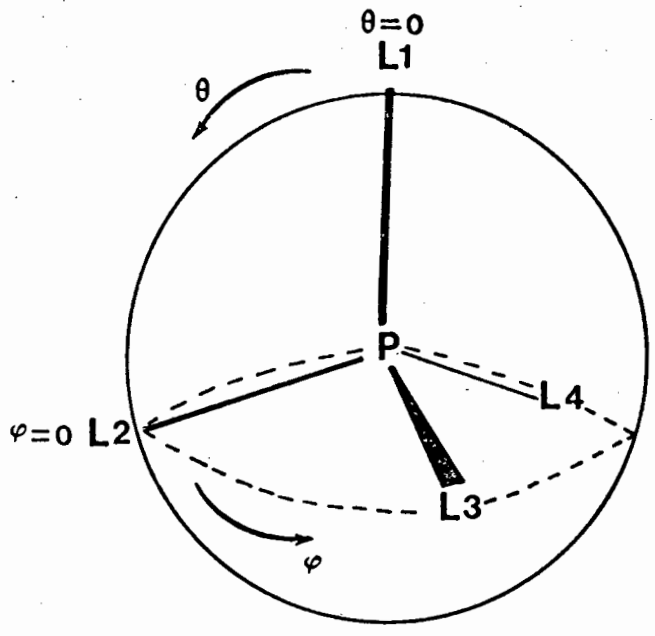


Figure 13.2 Definition of the spherical coordinate system (refer to Appendix 3)

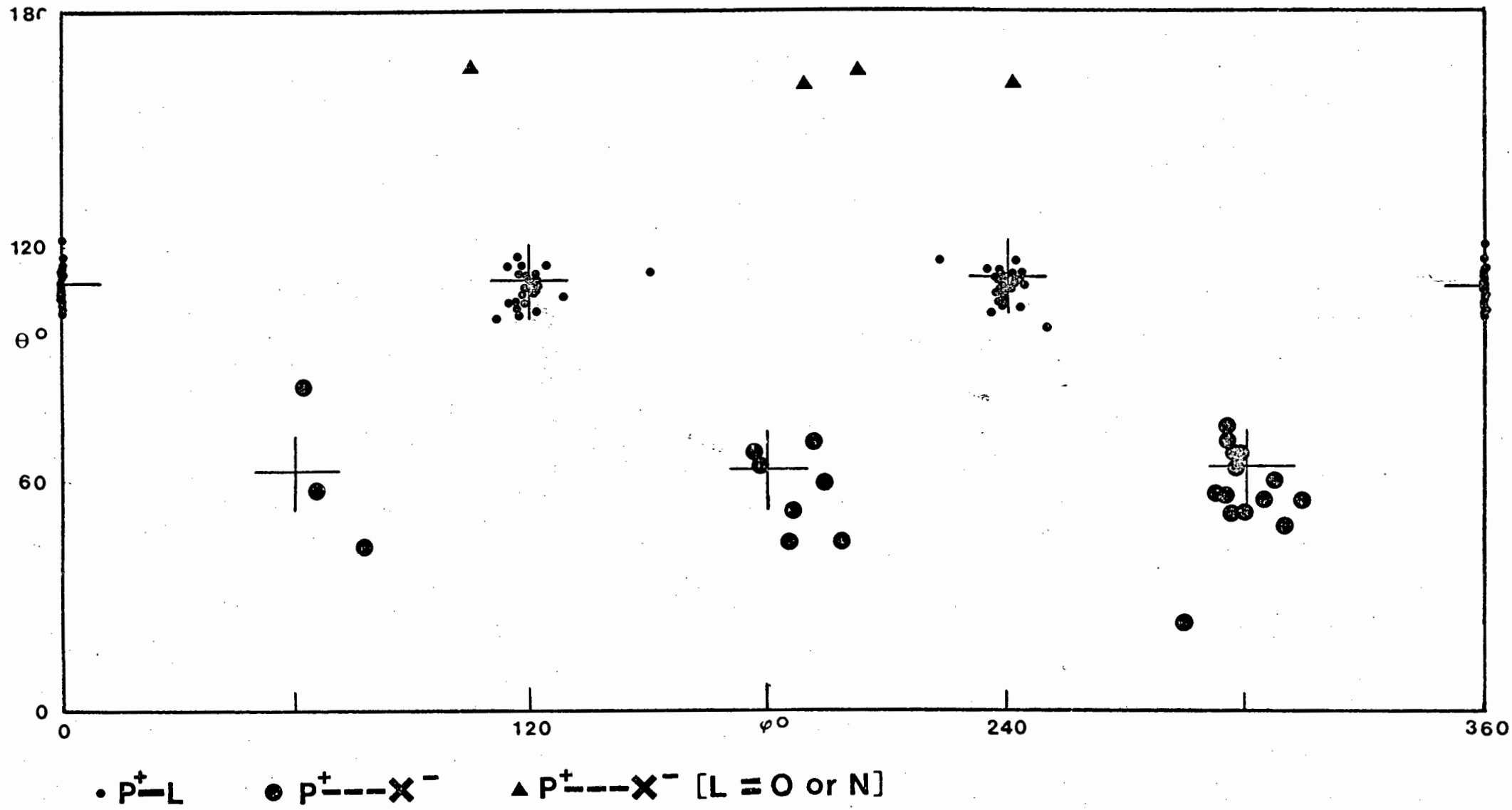


Figure 13.3 A plot of θ vs. ϕ - refer to the text

the absence of points in the region θ = 125.3°; ϕ = 120°, 240°, 360° ("edge" attack directions), it is clear how closely the nucleophiles favour the "face" approach.

The angular preference of the P^+----X^- directions shown in Figures 13.1 and 13.3 is the same as that obtained in the earlier study on the phosphonium ions[118] and similar to that found in the study of the approach of nucleophiles to sulphonium ions.[117] The P^+----X^- distances are considerably larger ($\Delta d > 0.5$ Å) than the corresponding S^+ ---- X^- distances ($\Delta d < 0.5$ Å). A probable reason for this difference is that sulphonium centers are sterically less hindered (only three ligands in the tetrahedral environment of sulphur) and thus more susceptible to the nucleophilic approach.

Compounds 1 to 19 in Figures 13.1 and 13.2 have one ligand attached to the phosphorus atom which can be considered both as a potential leaving group in substitution reactions (carbanion precursor) and as a precursor of an ylide, via α-hydrogen abstraction. In all these compounds the nucleophile approaches the phosphorus atom adjacent (i.e. on the same side) to the potential leaving group and not opposite it as expected in a S_N2 type substitution reaction. This tendency was also identified in the earlier study.[118] The anions of X^- are invariably located opposite an aromatic substituent, and this aversion of X^- to aromatic groups is possibly responsible for the deviations of the smaller L – P^+----X^- angles from the ideal "face" value of 70.5°. The nucleophile tends to "lean" away from the remaining aromatic substituents as a result of steric effects.

In the compounds where the potential leaving group has an acidic proton attached to an α-carbon atom (e.g. 1-19) an interaction between the approaching nucleophile X^- and the α-proton has been identified.[118] A similar interaction in the compounds 1-19 has also been found. The C_α----X^- distances for the compounds 1-19 are given in Table 13.2. In most of the compounds the C_α----X^- distances are only slightly greater than the corresponding sums of the van der Waals radii. In fact the C_α----X^- interaction is much stronger than the P^+----X^- interaction. The corrected C_α----X^- values are much closer to the accepted values for secondary bonds.

This interaction between the leaving group and the nucleophile X^- suggests that these compounds might be considered as models for ylide formation from a phosphonium ion and a base:

$$L_3P^+ - \underset{\displaystyle H\curvearrowleft X^-}{\underset{|}{C_2}}(R)_2 \rightleftharpoons L_3P = C(R)_2 + HX$$

This reaction should result in a change in the $P^+ - C_\alpha - R$ angle. In the compounds 1 to 16 the leaving group is the benzyl group and the $P^+ - C_\alpha - C(Ph)$ angle should change from the tetrahedral value to the planar trigonal value. The $P^+ - C_\alpha - R$ angles are listed in Table 13.2 and the enlargement of these angles over the expected sp^3 value is apparent. Therefore the observed proximity of anions X^- and electron-deficient α-carbon atoms observed in the earlier study of these compounds[118] is of a general nature and results from the weak hydrogen bonding of the type $P^+ - C - H$ ---- X^-, analogous to that postulated for the quarternary ammonium salts from infrared spectroscopic data[158]. Since the secondary bonds between the α-carbon atoms and the nucleophiles are long the first 19 compounds can only be considered for models

Table 13.2 Interactions $C_\alpha.....X^-$ vs. $P^+.....X^-$

Compound	$C_\alpha...X$	d	Δd	P...X	d	Δd	$P-C_\alpha-C$
BPPNCD (1)	3.67	3.45	0.22	4.44	3.55	0.89	115.5
	3.69	3.45	0.24	4.39	3.55	0.84	112.7
BZPHRU (2)	4.15	3.45	0.70	4.63	3.55	1.08	113.9
MPTCOB (3)	-*	-*	-*	3.73	3.65	0.08	115.6
PBPCCD (4)	4.00	3.45	0.55	4.45	3.55	0.90	114.2
	3.66	3.45	0.21	4.53	3.55	0.98	112.0
BZPPRU (5)	3.69	3.45	0.24	4.39	3.55	0.84	117.4
PBPBSN (6)	3.67	3.45	0.22	4.69	3.55	1.14	116.3
(8)	3.96	3.68	0.28	4.64	3.78	0.86	114.7
BMPPHB (9)	3.64	3.55	0.09	4.59	3.65	0.94	113.2
BZTPPI (10)	4.39	3.68	0.71	4.94	3.78	1.16	114.7
TBPZPU (11)	4.00	3.45	0.55	4.80	3.55	1.25	117.2
MPBPBR (12)	3.92	3.55	0.37	4.22	3.65	0.57	113.6
BZMNPB (13)	3.96	3.55	0.41	4.25	3.65	0.60	112.8
MOCTPB (14)	3.66	3.55	0.11	4.44	3.65	0.79	110.0
OMBPPB (15)	3.66	3.55	0.11	4.36	3.65	0.71	112.2
HTPOPY (16)	4.50	3.55	0.95	4.15	3.65	0.50	115.7
TPBOME (17)	4.20	3.55	0.65	4.45	3.65	0.80	111.4
							112.0
TPMYPB10 (18)	3.94	3.55	0.39	4.82	3.65	1.17	128.2
TPHDFC (19)	3.30	3.17	0.13	4.33	3.27	1.06	109.9
							111.1

For an explanation of the terms d, Δd etc refer to Appendix 2

*No close contacts

for the early stage of the ylide formation above.

We now consider the four compounds 20, 21, 22 and 24 (refer again to Appendix 2) where the leaving groups are of the form -OR or $-NR_2$. In all four of these compounds the pattern of L – P^+----X^- angles is consistent with "face" attack of the nucleophile. However now the nucleophile X^- approaches in a direction opposite to a potential leaving group. This is in direct contrast to what has been observed in the compounds discussed above. In these compounds there is no possibility of any interaction between the leaving group and nucleophile and the nucleophile approaches on the opposite side of the molecule. The P^+----X^- distances are still rather long, because the incoming nucleophile is sterically hindered by bulky groups. Therefore if these four compounds can be considered as models for the S_N2 type displacement at the phosphorus atom they then represent an 'early' stage along the reaction pathway. Presumably closer P^+----X^- bonds are only achieved by greater distortion of the P^+ tetrahedron.

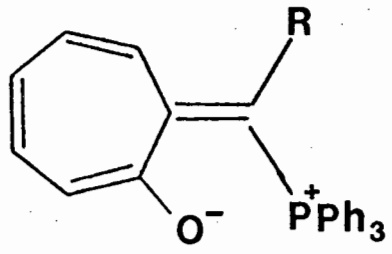
Compounds28, 29 and 30 are examples of compounds where the interaction of the nucleophile with the phosphorus atom is an intramolecular process. Significantly the P^+----X^- distance is less than the sum of the van der Waals radii and results in a much stronger interaction than that observed previously. Although many compounds were found in the CCDC file which exhibited a close intramolecular P^+----X^- interaction only those where the formation of a ring of five or more atoms was possible were considered chemically feasible. In the above three compounds the pattern of the P^+----X^- angles are consistent with "face" attack. The geometrical constraints of this intramolecular interaction result in nucleophilic attack adjacent to the expected leaving group.

The strong secondary P^+----O^- interactions in 29 and 30 result in significant distortions of the phosphonium ions (refer to Figure 13.4). In compound 29 the oxygen atom approaches the tetrahedral face defined by two phenyl groups and C(1). The $C - P^+ - C$ angles of the facial carbon atoms to the third phenyl group, which is opposite the approaching oxygen atom, are all much less than the tetrahedral angle of 109.5°. A similar situation is observed in compound 31. In addition the $C_f - P^+ - C_f$ angles, the angles involving the atoms on the face of the tetrahedron are enlarged over 109.5°. These distortions are characteristic of the distortion of a tetrahedron to form a trigonal bipyramid (see Figure 13.4). Although compound 28 also exhibits a strong P^+----N^- close contact it is a very crowded molecule and this crowding masks the distortions characteristic of the formation of a trigonal bipyramidal structure.

Table 13.3 Hydrogen Bonded Compounds

Compound	$\Delta d(P^+ \cdots X^-)$	$L - P^+ \cdots X^-$				H bond	Δd(H bond)
MHZPPB (23)	0.57	45.5	152.3	73.3	94.8	N-----Br	-0.12
TPOMBZ (25)	0.90	73.0	90.7	158.2	50.6	O----- N	0.12
HXTPPC (26)	0.31	34.5	83.5	138.0	104.8	O-----Cl	-0.52

Penultimately we consider the structures 23, 25 and 26. They are not listed in Figure 13.1 and the details of these three compounds are listed in Table 13.3. All these compounds exhibit an identifiable hydrogen bond between the expected leaving group and the approaching nucleophile.



29 , 30

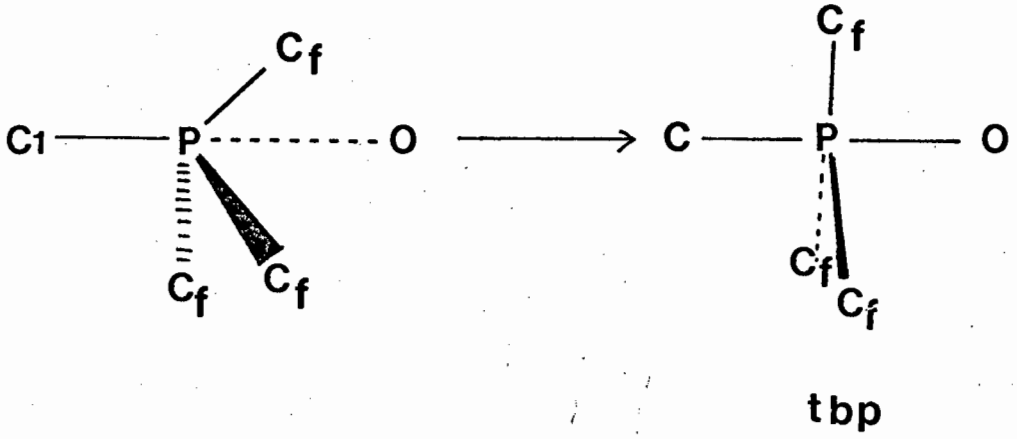


Figure 13.4 The intramolecular close contact in compounds 29 and 30 showing the formation of the trigonal bipyramidal (tbp) structure.

In these compounds there is a considerable scatter of the $L - P^+ - X^-$ angles about the directions associated with the "face" approach. As the strength of the hydrogen bond increases the distortion of the "face" angles increases. In the case of compound 26 the hydrogen bond is very short and the $L - P^+ \cdots X^-$ angles cannot be reconciled with the "face approach". It appears that in the cases where a favourable hydrogen bond can be formed the requirements of the hydrogen bond override the favourable "face" attack approach.

Finally, we consider the compounds 31 to 36 in which the phosphorus atom is substituted by phenyl, methyl or ethyl groups. The $C - P^+ \cdots X^-$ angles vs. Δd are shown in Figure 13.5. The striking feature of this plot is that despite the fact that the $P^+ \cdots X^-$ distances are very long the angular preferences found earlier are retained even at Δd values in excess of 3 Å. The $P^+ \cdots X^-$ distances are far too long to be considered as secondary bonds and the angular dependence is explained in terms of the requirements of close packing.[159] The closest packing of the phosphonium ion and the counter ion can be achieved by placing the counter ion between the ligands attached to the phosphorus atom - i.e. on the face of the tetrahedron.

The Geometry of the Phosphonium Ion

Table 13.4 gives the average values of the bond lengths and bond angles of the phosphonium ions discussed in this chapter. Generally there is little distortion of the angles around the phosphorus atom. In most of the compounds (except where there is an intramolecular interaction)

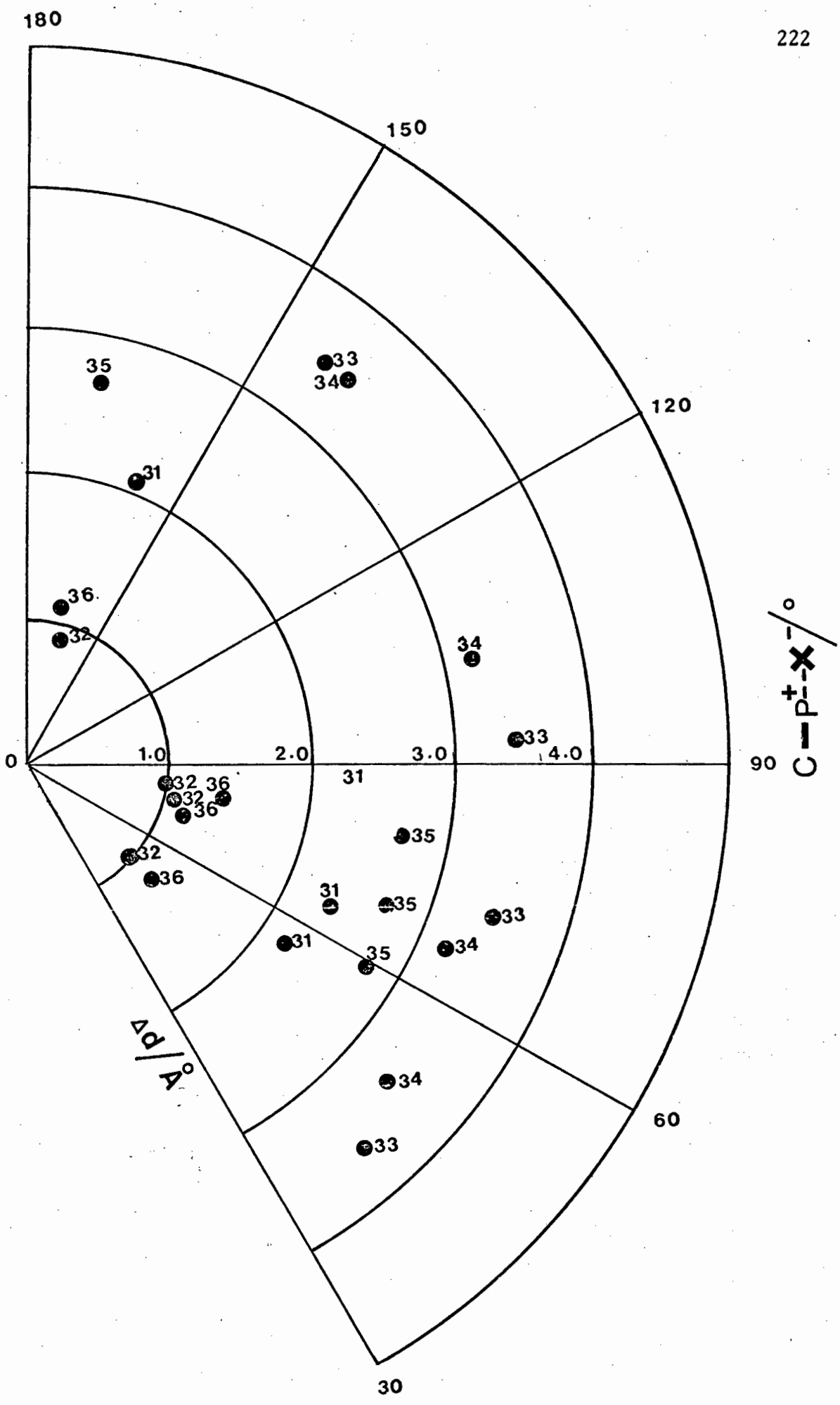


Figure 13.5 A plot of the distances Δd vs. the C — P^+ --- X^- angles for the compounds 31 to 36.

the approaching nucleophile is too far away to have an appreciable influence on the geometry of the phosphonium ion. The orientations of the groups attached to the phosphorus atom appear to be governed largely by packing forces[160].

Figure 13.6 shows a plot of θ, defined in Figure 13.2 against the mean of the angles $L_2 - P^+ - L_3$, $L_2 - P^+ - L_4$ and $L_3 - P^+ - L_4$. This plot simply gives an idea of the distortion of the $L - P^+ - L$ angles (ψ). This plot shows that most of the compounds are scattered about the mean values of $\theta = \psi \sim 109°$ expected in a sp^3 hybridized phosphorus. Compound three shows severe distortion of the $C - P - C$ angles and this is the result of the restriction in the $C - P^+ - C$ ring angle resulting in the distortion of the other angles. Compound 18 also shows large $C - P - C$ angle distortions and this is probably the result of the double bond character of the $P - CH - P^+$ bond in this compound.
It is significant that there are a number of compounds which deviate strongly from the linear curve which includes most of the compounds. The deviation of compound 17 is probably the result of an intermolecular interaction between the phosphorus atom and one of the oxygen atoms on the leaving group (the P^+----O = C distances are 2.94 Å and 3.32 Å). Compounds 29 and 30 both exhibit a strong intramolecular P^+----X^- close contact which leads to significant angular distortion. Compound 26 exhibits a strong O----Br hydrogen bond which appreciably distorts the $L - P^+$----X^- angles, indicating that the nucleophile does not approach along the pure "face" direction. Since the "face" approach is favoured in terms of the steric hindrance encountered by the approaching nucleophile, the "off face" approach adopted by the Br^- atom in compound 26 is expected to result in greater steric hindrance and hence greater distortion of the $L - P^+ - L$ angles on close approach. This is another

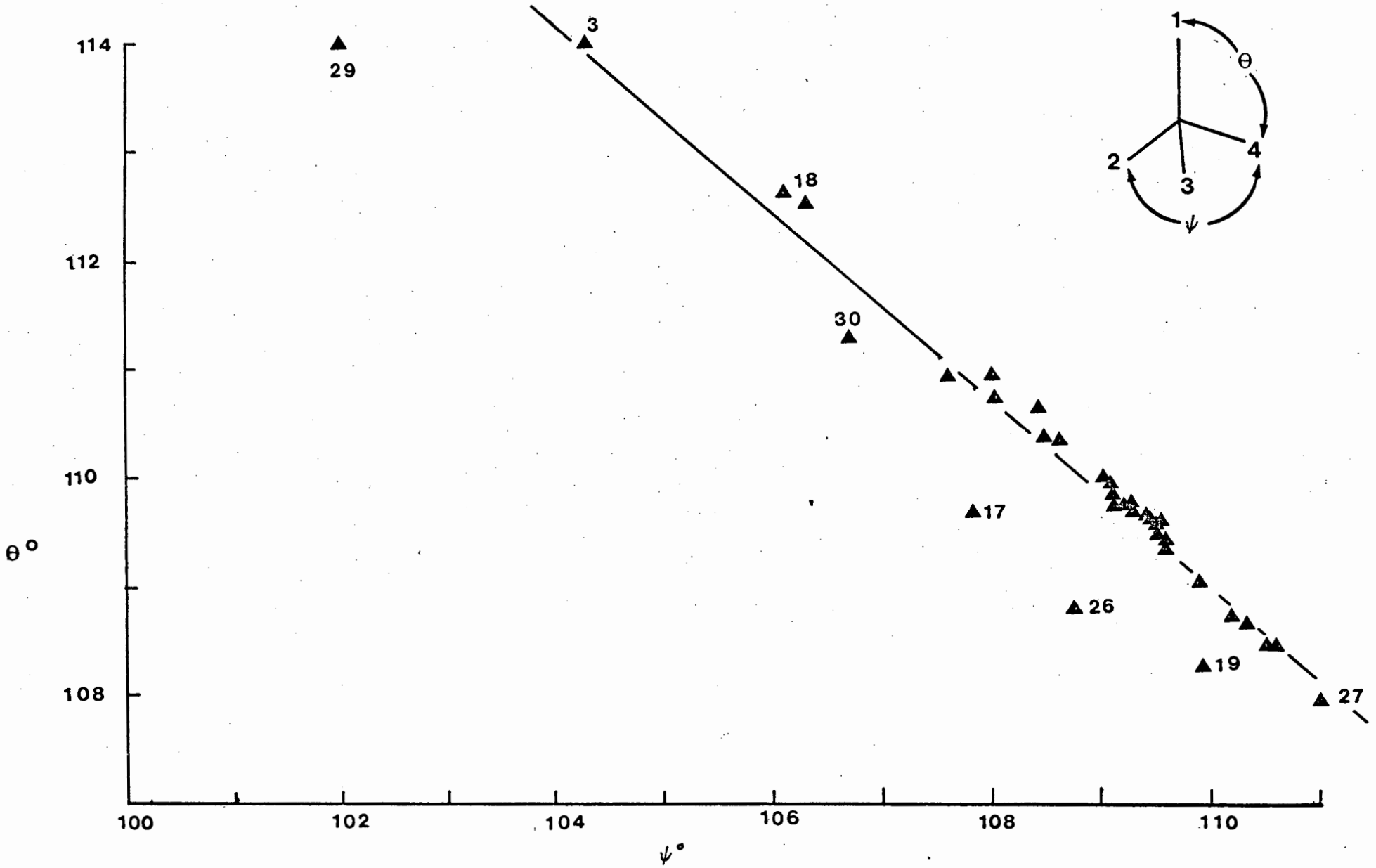


Figure 13.6 A plot of the angles θ vs. ψ for the phosphonium ions 1 - 36. For clarity selected compounds have been identified.

Table 13.4 Average Values of the Structural Parameters for the Phosphonium Ions

d(P – C)Å	d(P – O)Å	d(P – N)Å
1.774	1.559	1.659

∠(L – P – L)°
109.3

situation where the requirements of hydrogen bonding are achieved at the expense of other interactions.

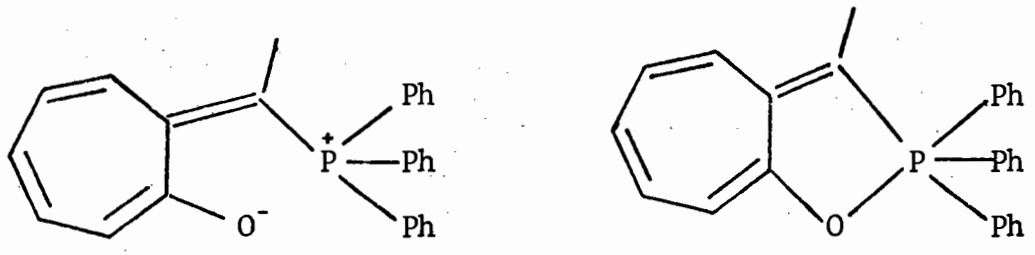
It therefore appears that the obvious distortions of some of the compounds above are mainly the result of repulsions between bulky groups attached to the phosphorus atom or packing factors. In the cases where there is close contact between the P^+ atom and X^- there is some distortion which is attributable to the approach of X^-. In the phosphonium ions studied this only occurs in the two compounds (29 and 30) where an intramolecular close contact is found. In a third compound there is some distortion as a result of the hydrogen bonding mode.

13.3 Conclusion

In all the compounds studied there is a preference for "face" approach of the nucleophile. This directional preference appears to be a

consequence of the requirements of packing rather than anything else. The "face" approach however, permits the close approach of the nucleophile to a potential α-proton on a bulky group attached to the phosphorus atom in the early stages of ylide formation. This approach also permits the nucleophile to approach the phosphorus atom opposite to a leaving group in a S_N2 type displacement reaction.

Although some compounds appear to behave as models for the S_N2 type displacement reaction the P^+----X^- distances are still rather long and more compounds are required for analysis. Unfortunately not many compounds of the type $RO - P^+ - R_3$ are available in the CCDC file[161]. In the two compounds where a close P^+----X^- contact is achieved geometrical constraints limit the approach options. Compounds 29 and 30 are considered as intermediates in the formation of the trigonal bipyramidal triphenyl phosphonium betaine (I).



(I)

In the case of the phosphonium ions the "Structural Correlation Principle" given has fresh insight into the dynamics of these compounds. In the case of the compounds where ylide formation is proposed the evidence is fairly convincing. On the other hand, the limited number of structures where other mechanisms are believed to operate, must be approached more cautiously.

APPENDIX ONE

A1. THE STRUCTURE OF SYNTHETIC HYDRATED AND DEHYDRATED ANALCIME

A1.1 Introduction

Analcime, $NaAlSi_2O_6.H_2O$, is an alkali aluminosilicate and an example of a zeolite[166,167] which occurs fairly widely in rocks[168]. A feature of zeolite structures is the presence of cavities interconnected by apertures which permit the passage of water molecules or other molecules from one cavity to the next. These cavities and apertures are bounded by $(Al,Si)O_4$ tetrahedra. The water molecules in the cavities are loosely bound and can be driven off on heating. When the water is removed, it can be replaced by other molecular groups such as the atmospheric gases, ammonia or alcohol.

The cavities also contain positive ions, generally alkali metal or calcium ions, the charges of which balance the negative charge of the framework. These positive ions may be exchanged by other positive ions. For example the sodium ions may be exchanged by calcium ions or vice-versa. The groups that can replace water or be exchanged for the positive ions are limited by the size of the apertures or 'windows' giving access to the cavities. This property of the zeolites to permit groups below a certain size to pass, while refusing passage to larger groups, has led to their being termed 'molecular sieves'.

Analcime is an example of a small-pore zeolite - i.e. the size of the windows that permit the passage of molecules into the cavities limits its usefulness as a molecular sieve. Our interest in zeolites is part of a program to study the structures of zeolites exchanged with various

species. In particular we were investigating the absorption of phosphorus by zeolite A and zeolite X with a view to determining the structures (by single crystal methods) of the phosphorus loaded zeolites. The pore size of analcime prevents the absorption of phosphorus atoms but it was chosen as a starting point in the study of zeolite structures because crystals were available. We were however unable to proceed with the study because we were unable to obtain crystals of suitable size of the zeolite A and X. The structure of analcime is however reported here as it afforded a valuable opportunity to gain experience of the determinations of structures of high symmetry.

The crystal structures of analcime and its Ca^{2+} and K^{+} exchanged analogues having been repeatedly studied[169-175]. Although refinement was generally carried out in the cubic space group *Ia3d*, the possibility of lower symmetries is suggested by optical anomalies and 'forbidden' X-ray reflections. This has been elegantly demonstrated by Mazzi and Galli[176], who have identified both orthorhombic and tetragonal forms. This is the first synthetic analcime to be studied by X-ray diffraction.

Zeolite structure analysis is deceptive because it poses several problems and, to quote Karl Fischer[177]: "Zeolites, unfortunately, are in most cases not normal but 'pathological' crystals in many respects; equipoints for H_2O and cations may not be fully occupied, and the correspondingly small electron-density peaks cannot always be clearly labelled for a specified atom".

A1.2 Experimental

Analcime crystals were synthesized with the aid of an autoclove[178,179]. 87.1 g of sodium aluminate ($Na_2O.Al_2O_3.3H_2O$) was dissolved in 275 ml of water and stirred and after the solid had dissolved 242.6 g of a 40 % SiO_2 / 60 % H_2O mixture (ca. 180 ml) was added slowly and stirred for 1 hour. This solution was then heated to 90°C while stirring until the volume had reduced to ca. 250 ml and pH ∿ 14. To this gel a solution of 3.2 g NaOH in ca. 50 ml was added while stirring. This gel was then placed in an autoclave and maintained at 180 - 200°C for five days.

The resulting crystals were remarkably homogenous in size, exhibited a regular trapezohedral habit, had average diameters of 125 μm and were optically isotropic. A photograph of some of the analcime crystals is shown in Figure A1.1.

Dehydration of the crystals was carried out in the cell shown in Figure A1.2. Single crystals were placed in glass capillaries and fused to the base of the cell C. The desorption process was followed by the McBain balance technique[180,181], placing a suitable quantity of sample in the glass bucket A suspended at the end of the silica spring B. The dehydration cell C was maintained at a pressure of less than 1 mm Hg and 350°C for 24 hours (heater at D). The loss of water from the sample was monitored by observing the shortening of the spring with the travelling telescope at E. The spring had previously been calibrated. After 24 hours the capillaries at the base of the cell were sealed under vacuum and removed for single crystal diffraction. The change in weight of the sample indicated a weight loss of 8.03%. Based on the ideal stoichiometry for analcime ($NaAlSi_2O_6.H_2O$) total loss of H_2O represents a weight change of 8.1 %. The dehydrated crystals were opaque but retained the same morphology as the hydrated analogue.

The crystal data as well as the details of the diffractometer data collection are given in Table A1.1. The diffractometer data were consistent with the cubic space group $Ia3d$[11] as found previously in other cubic forms of analcime.

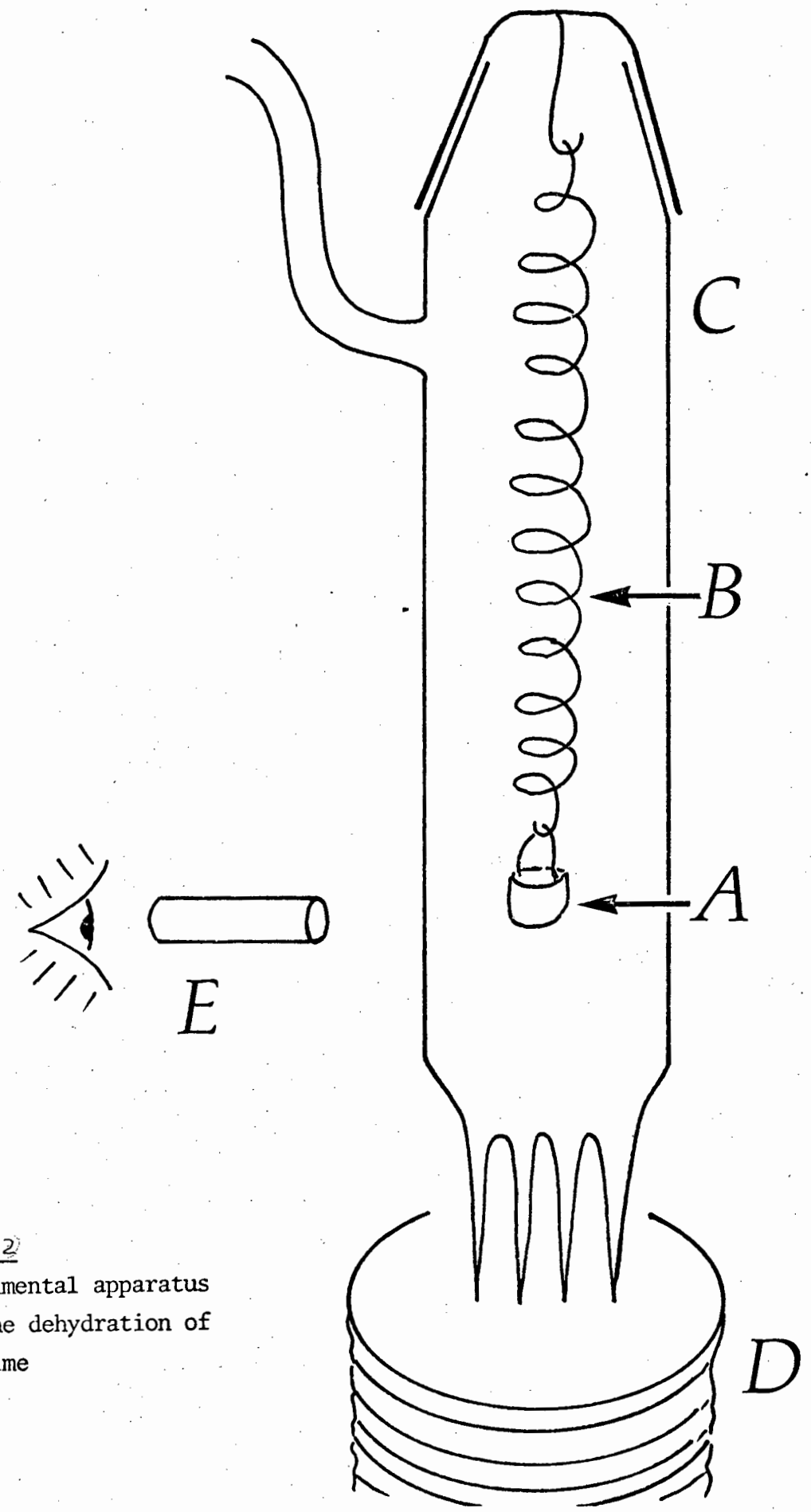


Figure A1.2
The experimental apparatus used in the dehydration of the analcime

Table A1.1 Crystal and Intensity Data and Refinement Parameters for the Structures

Crystal Data	Hydrated analcime	Dehydrated analcime
Space group	*Ia3d*	*Ia3d*
a	13.710(7) (Å?)	13.631(7) Å
u	2576.99 Å^3	2532.70 Å^3
μ(Mo-K_α)	6.52 cm^{-1}	6.48 cm^{-1}
F(000)	1746	1586
Crystal dimensions	1.75x1.75x1.75 mm	1.75x1.75x1.75 mm
Scan width	1.4° θ	1.4° θ
Scan speed	0.03° θ s^{-1}	0.03° θ s^{-1}
Range of reflections	4<θ<30°	4<θ<30°
Number of unique reflections	406	304
Observed reflection criteria	$I_{rel} > 2\sigma I_{rel}$	$I_{rel} > 2\sigma I_{rel}$
Number of reflections observed	240	188
Number of variables	20	18
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.031	0.084
$R_w = \Sigma w^{\frac{1}{2}}\|\|F_o\|-\|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$ where $w = (\sigma^2 F)^{-1}$	-	0.062

A1.3 Solution and Refinement of the Structures

In the hydrated analcime the atomic parameters of Ferraris *et al.*[172] were used as starting coordinates. All refinements were carried out with the SHELX[12] program system. Initially we were unable to refine the position of the oxygen atom as published by Ferraris *et al.* and it appears that there is an error in the published atomic positions for this atom. The position of the oxygen atom was located in a difference electron-density map. It is interesting to note how the SHELX system was employed for placing atoms at special positions. Thus the Si/Al atom which is located at Wykoff position *g* ($\frac{1}{8}$, x, $\frac{1}{4} - x$) could not be placed at the position $\frac{1}{8}$, x, $\frac{1}{4} - x$ because of the way the coordinates are related in terms of free variables (FVAR) in the SHELX[12] system. Therefore the position of the Si/Al atom was placed at the symmetrically related position $-x$, $\frac{3}{4} + x$, $\frac{7}{8}$ which is input in SHELX as:

x	y	z
19.75	-19.75	10.875

These positions are referred to free variable number two and are interpreted by the program as follows:

$$19.75 \equiv -0.25(4x)$$

$$-19.75 \equiv -0.25(1-4x) = -0.25 + x \equiv 0.75 + x$$

$$10.875 \equiv 0.875$$

where free variable two is given the value of $4x$. The positions of the Si and Al atoms were tied together and their site occupancy factors fixed so as to take into account the required Si/Al ratio. The scattering factors of the Na and Al atoms were taken from the International Tables for Crystallography[11]. The scattering factors used for Si were those utilized by SHELX[12].

After a number of cycles of least-squares refinement with all the atoms treated anisotropically[182], the *R* factor converged to 4.01 %. These refinements were based on an ideal Si/Al ratio of 2:1. The analcime crystals were subjected to an elemental analysis in an electron microprobe. The results of this analysis indicated a Si/Al ratio of 2.33 : 1. This analysis was confirmed by the analysis of the Si/Al – O distances according to the method of Jones[183,176] in which a plot of Si/Al – O distances is correlated with the Al fraction of the $(Si/Al)O_4$ tetrahedra in aluminosilicate framework structures. In the final least-squares refinement the Si/Al ratio was therefore adjusted to 2.33:1. In the final cycle of least-squares refinement the average shift to error ratio was less than 1 %. An analysis of variance calculated after the final refinement is given in Table A1.2. No hydrogen atoms were searched for and a difference electron-density map calculated after the final cycle of least squares refinement there were no peaks of height > 0.41 e $Å^3$. The details of the final refinement are listed in Table A1.1.

The dehydrated analcime was refined using the final coordinates obtained after the refinement of the hydrated analogue with water atoms omitted. After a number of cycles of least-squares-refinement a difference electron-density map was calculated which revealed a peak of 0.81 e $Å^3$ at the water oxygen site indicating that the analcime was not completely dehydrated. This nevertheless means that only a very small fraction of the original water molecules remain. Attempts to refine both the temperature factor and the site occupancy factor of this water oxygen failed, and it thus was omitted from the final least-squares refinement.

Table A1.2 Analysis of Variance[a] for Hydrated Analcime

(a) By parity groups

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	79	0	0	55	0	61	45	0	240
V	275	0	0	348	0	237	280	0	286

(b) As a function of sin (θ)

Sin(θ)	0	.21	.27	.31	.35	.38	.41	.43	.46	.48	.50
N	24	25	23	29	24	26	17	31	20	21	
V	555	164	225	192	146	336	230	241	290	268	

(c) As a function of $\sqrt{F/Fmax}$

F/Fmax	0	.19	.22	.24	.27	.29	.31	.32	.37	.43	1.00
N	28	24	20	32	25	24	17	23	25	22	
V	372	320	371	192	182	199	199	226	355	341	

(d) As a function of |Miller Index|

|h|	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	32	53	40	34	27	17	15	7	8	5	2	0	0	0	0
V	259	332	263	240	316	275	247	314	273	257	429	0	0	0	0

|k|	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	3	5	20	18	30	20	29	20	31	25	22	11	5	1	0
V	384	666	366	279	270	265	199	279	197	335	221	267	368	155	0

|l|	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	0	0	2	2	6	4	9	10	19	18	25	23	28	22	72
V	0	0	698	406	611	637	433	224	220	191	260	226	251	196	269

[a] N = Number of reflections in the group; $V = 100[M\Sigma(w|F_o - F_c|^2)/N\Sigma w]$; M = Total number of reflections.

An analysis of variance calculated after the final cycles of least-squares refinement is given in Table A1.3. Lists of observed and calculated structure factors for both structures are given in Tables A1.10 and A1.11. Final atomic coordinates for both compounds are given in Table A1.4.

A1.4 Description of the Structures and Discussion

The bond lengths and angles describing the Na octahedra, Si/Al tetrahedra and the channel apertures of both structures are listed in Tables A1.5 to A1.9. The Na octahedra, Si/Al tetrahedra and channel apertures are shown in Figures A1.3, A1.4 and A1.5 respectively. The estimated standard deviations in Tables A1.7 to A1.11 were calculated using the BONDLA subroutine of the X-RAY 76 program system[14].

The structure of the hydrated form of analcime is essentially the same as that reported previously (Ferraris *et al.*[172]). In our synthetic analcime there are 34 Si and 14 Al atoms randomly distributed over the 48 available sites. Each occupied Si/Al site is surrounded by two oxygen atoms (d = 1.641 Å) and a further two oxygens (d = 1.647 Å). These bond distances are similar to those reported by Ferraris *et al.* Each occupied Na site is surrounded by four oxygen atoms at a distance of 2.503 Å in a square planar arrangement. The Na site is also close to two possible water oxygen sites. Therefore each Na site has a possible octahedral environment of oxygen atoms. (Figure A1.3).

Table A1.3 Analysis of Variance[a] for Dehydrated Analcime

(a) By parity group

Group	GGG	UGG	GUG	UUG	GGU	UGU	GUU	UUU	ALL
N	60	0	0	44	0	48	36	0	188
V	546	0	0	576	0	461	416	0	510

(b) As a function of sin (θ)

sin(θ)	0	.20	.25	.29	.32	.36	.38	.41	.45	.48	.50
N	22	18	20	17	19	19	20	23	16	14	
V	819	434	523	436	403	355	505	432	467	509	

(c) As a function of $\sqrt{F/Fmax}$

$\sqrt{F/Fmax}$	0	.23	.24	.26	.27	.29	.30	.33	.38	.45	1.00
N	32	11	23	10	32	10	16	17	18	19	
V	541	362	474	599	392	328	543	460	535	721	

(d) As a function of |Miller Index|

||	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	29	45	31	23	19	13	11	9	5	2	1	0	0	0	0
V	631	588	456	381	452	470	413	547	265	382	9	0	0	0	0

||	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	3	4	17	15	27	19	23	15	22	20	13	6	3	1	0
V	1033	1112	617	525	507	388	401	515	409	447	478	196	600	825	0

||	0	1	2	3	4	5	6	7	8	9	10	11	12	13	REST
N	0	0	2	2	6	5	11	9	17	16	22	19	17	17	45
V	0	0	1536	468	1020	707	317	521	402	453	549	460	288	345	501

[a] N = Number of reflections in the group; $V = 100[M\Sigma(w|F_o-F_c|^2)/N\Sigma w]$; M = Total number of reflections.

Table A1.4 Fractional Atomic Coordinates ($\times 10^4$) and Anisotropic Temperature Factors ($Å^2 \times 10^3$) with e.s.d.'s in Parentheses.

Coordinates

	Hydrated			Dehydrated		
	x	y	z	x	y	z
O(1)	2197(1)	1047(1)	3658(2)	2199(3)	1037(3)	3657(1)
Si	-6622(1)	4122(1)	8750	-6623(1)	4123(1)	8750
Al	-6622(1)	4122(1)	8750	-6623(1)	4123(1)	8750
Na	1250	0	2500	1250	0	2500
W	1250	1250	1250			

Temperature factors

Hydrated

	U11	U22	U33	U23	U13	U12
O	18(1)	34(1)	38(1)	-6(1)	-2(1)	6(1)
Si	13(1)	12(1)	12(1)	5(1)	-1(1)	-1(1)
Al	13(1)	12(1)	12(1)	5(1)	-1(1)	-1(1)
Na	40(2)	43(2)	43(2)	-21(3)	0	0
W	82(3)	82(3)	82(3)	25(4)	25(4)	25(4)

Dehydrated

O	32(3)	60(4)	66(4)	-4(3)	-2(3)	11(3)
Si	28(3)	23(1)	23(1)	8(2)	-4(1)	-4(1)
Al	28(3)	23(1)	23(1)	8(2)	-4(1)	-4(1)
Na	146(13)	193(13)	193(13)	-140(16)	0	0

Site occupancy factors: O = 1.0000

Si = 0.3542 Al = 0.1458

Na = 0.1667 W = 0.1667

Table A1.5 Bond Lengths (Å) of the Na Octahedra with e.s.d.'s in Parentheses.

Bond	Hydrated	Dehydrated
Na(1)-O(1)	2.503(3)	2.482(4)
Na(1)-O(2)	2.503(3)	2.482(4)
Na(1)-O(3)	2.503(3)	2.482(4)
Na(1)-O(4)	2.503(3)	2.482(4)
W(1)-O(1)	3.558(3)	3.539(4) *
W(1)-O(2)	3.409(3)	3.378(4) *
W(1)-O(3)	3.558(3)	3.539(4) *
W(1)-O(4)	3.409(3)	3.378(4) *
W(2)-O(1)	3.409(3)	3.378(4) *
W(2)-O(2)	3.558(3)	3.539(4) *
W(2)-O(3)	3.409(3)	3.378(4) *
W(2)-O(4)	3.558(3)	3.539(4) *
Na(1)-W(1)	2.424(1)	2.410(1) *
Na(1)-W(2)	2.424(1)	2.410(1) *
O(1)-O(2)	2.606(4)	2.597(6)
O(3)-O(4)	2.606(4)	2.597(6)
O(1)-O(4)	4.281(4)	4.236(6)
O(2)-O(3)	4.281(4)	4.236(6)

* These bonds refer to the distance from the vacant water site

Table A1.6 Principal Bond Angles (°) of the Na Octahedra with e.s.d.'s in Parentheses.

Angle	Hydrated	Dehydrated
W(1)-Na(1)-W(2)	180.0(1)	
O(1)-Na(1)-O(2)	62.7(1)	63.1(1)
O(3)-Na(1)-O(4)	62.7(1)	63.1(1)
O(2)-Na(1)-O(3)	117.5(1)	117.2(1)
O(1)-Na(1)-O(4)	115.5(1)	117.2(1)
W(1)-Na(1)-O(1)	92.5(1)	
W(1)-Na(1)-O(3)	92.5(1)	
W(1)-Na(1)-O(2)	87.5(1)	
W(1)-Na(1)-O(4)	87.5(1)	
W(1)-O(1)-W(2)	88.1(1)	
W(2)-Na(1)-Na(2)	150.0(1)	
O(1)-W(1)-O(3)	89.3(1)	
O(2)-W(1)-O(4)	94.9(1)	
O(1)-W(1)-O(4)	75.8(1)	
O(2)-W(1)-O(3)	75.8(1)	
O(3)-W(1)-O(4)	43.9(1)	
O(1)-W(1)-O(2)	43.9(1)	
Na(1)-Na(2)-Na(3)	60.0(1)	60.0(1)

Table A1.7 Bond Lengths (Å) describing the Si/Al Coordination Site with e.s.d.'s in Parentheses.

Bond	Hydrated	Dehydrated
Si/Al-O(13)	1.641(2)	1.625(4)
Si/Al-O(15)	1.461(2)	1.625(4)
Si/Al-O(14)	1.647(2)	1.648(5)
Si/Al-O(16)	1.647(2)	1.648(5)
O(13)-O(15)	2.630(2)	2.705(6)
O(13)-O(15)	2.630(2)	2.592(6)
O(13)-O(14)	2.708(2)	2.705(6)
O(13)-O(16)	2.725(2)	2.716(7)
O(15)-O(14)	2.725(2)	2.716(2)
O(15)-O(16)	2.708(2)	2.705(6)
O(14)-O(16)	2.606(2)	2.592(8)

Table A1.8 Bond Angles describing the Si/Al Coordination Site with e.s.d.'s in Parentheses

Angle/°	Hydrated	Dehydrated
O(13)-O(14)-O(15)	57.9(1)	57.1(2)
O(13)-O(14)-O(16)	61.7(6)	61.6(2)
O(13)-O(15)-O(14)	60.7(1)	61.2(2)
O(13)-O(15)-O(16)	61.37(1)	61.7(2)
O(13)-O(16)-O(15)	57.9(1)	57.1(2)
O(13)-O(16)-O(14)	61.0(1)	61.2(2)
O(14)-O(13)-O(16)	57.3(1)	57.3(2)
O(14)-O(13)-O(15)	61.4(1)	61.7(2)
O(14)-O(15)-O(16)	57.3(1)	57.3(2)
O(14)-O(16)-O(15)	61.7(1)	61.6(2)
O(15)-O(13)-O(16)	60.7(1)	61.2(2)
O(15)-O(14)-O(16)	61.0(2)	61.2(2)
O(13)-Si/Al-O(15)	106.5(1)	105.7(2)
O(13)-Si/Al-O(14)	110.9(1)	111.5(2)
O(15)-Si/Al-O(14)	112.0(1)	112.1(2)
O(13)-Si/Al-O(16)	112.0(1)	112.1(2)
O(15)-Si/Al-O(16)	110.9(1)	111.5(2)
O(14)-Si/Al-O(16)	104.6(1)	104.0(2)

Table A1.9 Bond Lengths (Å) and Angles describing the Six-membered Channel apertures with e.s.d.'s in Parentheses.

Bond/Angle	Hydrated	Dehydrated
O(1)-O(7)	2.708(2)	2.705(6)
O(7)-O(5)	2.708(2)	2.705(6)
O(5)-O(8)	2.708(2)	2.705(6)
O(8)-O(6)	2.708(2)	2.705(6)
O(6)-O(9)	2.709(2)	2.705(6)
O(9)-O(1)	2.708(2)	2.705(6)
O(1)-O(8)	5.162(3)	5.151(6)
O(7)-O(6)	5.162(3)	5.151(6)
O(5)-O(9)	5.162(3)	5.151(6)
W-O(1)	3.558(2)	3.539(4)
W-O(7)	4.276(2)	4.262(4) *
W-O(5)	3.558(2)	3.539(4) *
W-O(8)	4.276(2)	4.262(4) *
W-O(6)	3.558(2)	3.539(4) *
W-O(9)	4.276(2)	4.262(4) *
O(1)-O(7)-O(5)°	108.5(1)	108.2(2)
O(7)-O(5)-O(8)°	108.5(1)	108.2(2)
O(5)-O(8)-O(6)°	108.5(1)	108.2(2)
O(8)-O(6)-O(9)°	108.5(7)	108.2(2)
O(6)-O(9)-O(1)°	108.5(1)	108.2(2)
O(9)-O(1)-O(7)°	108.5(1)	108.2(2)

These distances refer to the distances from the vacant water site in the dehydrated compound.

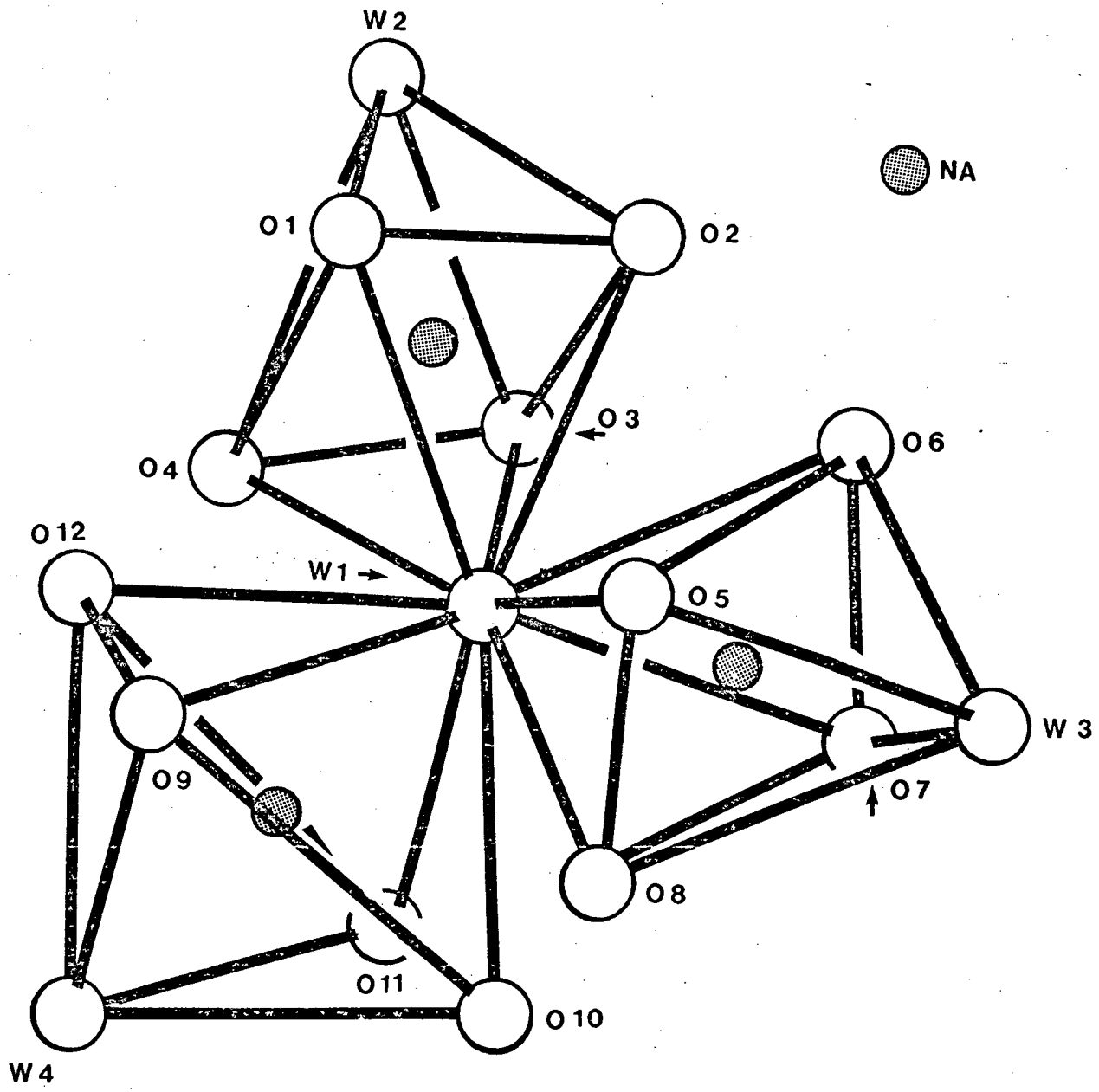


Figure A1.3 A view of the Na coordination site

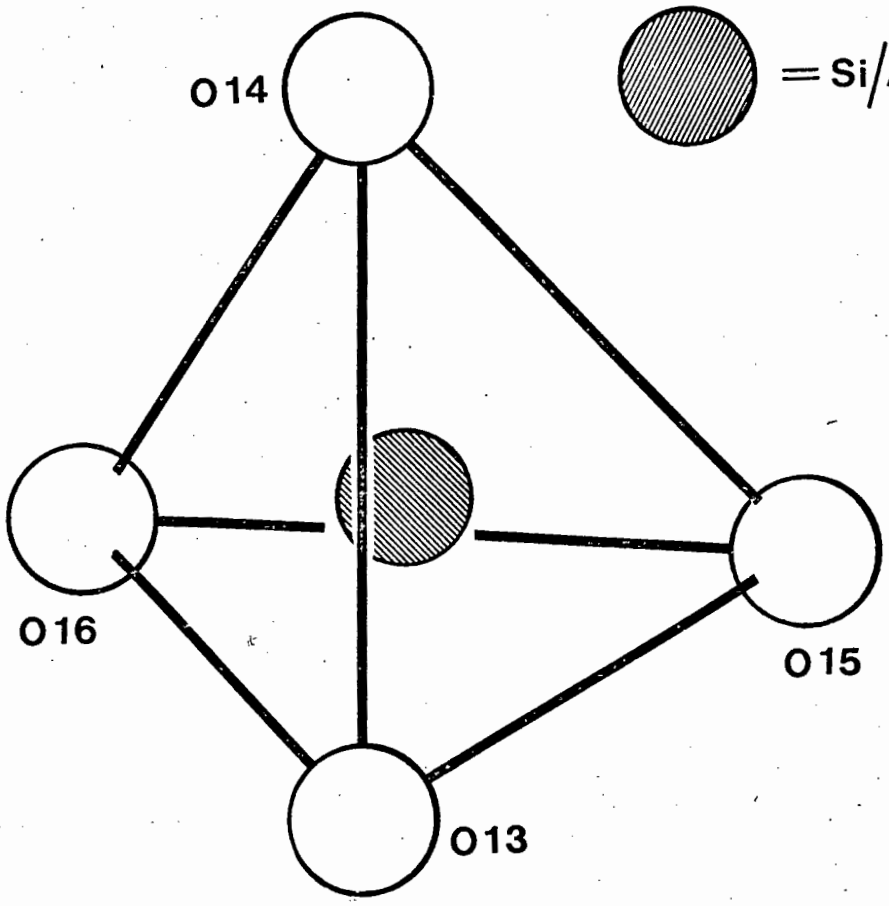


Figure A1.4 A view of the Si/Al coordination site

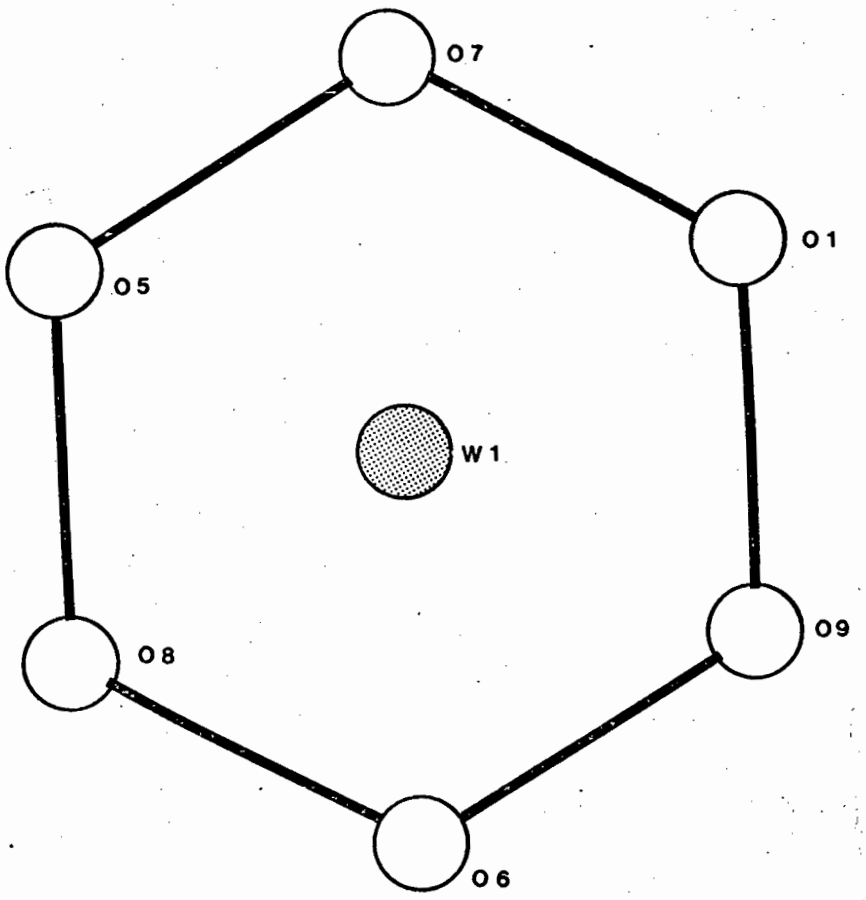


Figure A1.5 A view of the six-membered cavity windows

The replaceable H_2O molecules in the analcime are bound in non-intersecting channels which lie along the three-fold axes. The apertures of these channels are bounded by oxygen atoms of connected tetrahedra. In the case of analcime the apertures involve six oxygen atoms giving rise to a six membered ring. This is shown in Figure A1.5. The size of this ring is a measure of the channel size in analcime. For the hydrated analcime, the ring diameter (defined as the internuclear distance between any pair of opposite oxygen atoms) = 5.162 Å, which leaves a 'channel size' of 5.16 - 2(1.4) = 2.36 Å, when we take into account the van der Waals radius of oxygen = 1.4 Å. On loss of water, there is a shrinkage of the unit cell length by ≈ 0.6 % (Table A1.1) and the Na^+ ion exhibits enhanced thermal motion.

In the dehydrated form the Na-O distances are 2.482 Å compared to 2.503 Å in the hydrated form. In addition the Na atom is displaced 0.014 Å towards the vacant water site in comparison to the hydrated compound. It appears therefore that the coordination of Na in the dehydrated analcime is similar to that in the hydrated form if the water oxygen is ignored.

The Si/Al coordination in the dehydrated form is also little changed from that observed in the hydrated forms. The Si/Al – O distances in the hydrated form are 1.641 and 1.647 Å and in the dehydrated form 1.625 and 1.648 Å. Likewise the diameter of the six-membered aperture cavities in the dehydrated form is 0.011 Å smaller than that found in the hydrated form. This value is too small to significantly change the size of the molecules which are absorbed in the gas phase since these processes occur at elevated temperatures.

Table A1.10 Structure Factors for Hydrated Analcime

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	1	2	162	172	0	2	8	104	103	2	9	9	100	-100	5	6	11	42	41	2	10	12	36	35
0	2	2	116	-119	2	2	8	63	-63	6	9	9	60	59	2	7	11	47	-46	4	10	12	41	40
1	2	3	50	-55	1	3	8	127	126	1	1	10	178	-178	1	8	11	114	114	6	10	12	31	-33
2	3	3	269	267	0	4	8	35	-34	0	2	10	70	-70	3	8	11	36	37	10	10	12	20	24
0	0	4	632	-629	2	4	8	93	93	1	3	10	106	-104	5	8	11	88	-89	1	11	12	24	-22
0	2	4	15	-9	4	4	8	64	-65	3	3	10	138	139	2	9	11	33	-33	7	11	12	23	19
2	2	4	91	90	1	5	8	29	-26	0	4	10	73	73	4	9	11	42	-42	9	11	12	19	-15
1	3	4	113	-110	3	5	8	63	-63	2	4	10	53	51	6	9	11	16	9	0	12	12	48	-48
0	4	4	170	169	0	6	8	161	-164	1	5	10	114	117	8	9	11	60	62	4	12	12	17	-13
4	4	4	34	-47	2	6	8	36	35	3	5	10	85	-87	1	10	11	34	-35	8	12	12	64	68
1	2	5	112	-122	4	6	8	28	29	5	5	10	158	-165	3	10	11	48	-49	1	2	13	46	-43
2	3	5	74	-67	6	6	8	93	-93	0	6	10	53	53	5	10	11	17	-16	1	4	13	60	-60
1	4	5	36	-35	1	7	8	153	-154	4	6	10	22	-23	7	10	11	71	70	3	4	13	25	-26
3	4	5	40	40	3	7	8	56	53	1	7	10	57	56	9	10	11	23	-20	4	5	13	56	-57
1	1	6	102	-91	5	7	8	99	99	3	7	10	27	-31	2	11	11	53	50	1	6	13	50	51
0	2	6	56	52	0	8	8	48	52	0	8	10	97	-101	10	11	11	44	-48	3	6	13	38	39
1	3	6	48	-46	4	8	8	34	33	2	8	10	47	-44	0	2	12	64	-64	6	7	13	35	33
3	3	6	153	-155	8	8	8	71	70	4	8	10	53	51	2	2	12	38	40	1	8	13	64	61
0	4	6	219	-221	1	2	9	106	105	6	8	10	29	30	1	3	12	43	-45	3	8	13	21	22
2	4	6	37	-33	2	3	9	140	-139	1	9	10	45	-44	0	4	12	43	-43	5	8	13	62	-62
1	5	6	106	106	1	4	9	36	34	3	9	10	56	-58	2	4	12	61	-60	2	9	13	67	64
0	6	6	141	-142	3	4	9	20	-18	5	9	10	87	91	1	5	12	24	24	4	9	13	25	24
4	6	6	101	101	2	5	9	40	-38	7	9	10	60	66	0	6	12	166	166	6	9	13	30	-29
1	2	7	59	-59	4	5	9	28	29	9	9	10	118	-119	2	6	12	32	-33	8	9	13	27	-22
2	3	7	180	-176	1	6	9	102	-104	0	10	10	57	-58	4	6	12	88	-88	3	10	13	39	-37
1	4	7	131	129	3	6	9	147	-151	4	10	10	27	24	6	6	12	79	77	5	10	13	39	-41
3	4	7	44	46	5	6	9	42	43	8	10	10	25	-25	1	7	12	50	51	7	10	13	70	69
2	5	7	19	-20	2	7	9	36	41	1	2	11	72	-76	5	7	12	17	-10	9	10	13	44	43
4	5	7	77	-77	4	7	9	49	49	2	3	11	24	22	0	8	12	46	46	2	11	13	77	-77
1	6	7	50	51	6	7	9	66	-67	1	4	11	86	-85	4	8	12	21	-20	6	11	13	38	35
3	6	7	98	101	1	8	9	38	-38	3	4	11	24	-23	6	8	12	42	-45	8	11	13	21	-23
2	7	7	233	231	3	8	9	25	27	2	5	11	48	48	8	8	12	47	-47	2	13	13	59	-60
6	7	7	131	-133	5	8	9	40	39	4	5	11	63	63	3	9	12	16	-16	1	1	14	113	111
0	0	8	335	329	7	8	9	58	-60	1	6	11	51	51	0	10	12	61	-65	1	3	14	63	64

Table A1.10 Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	3	14	67	-68	4	12	14	21	-15	8	9	15	24	-29	4	6	16	72	72	1	2	17	30	-31
0	4	14	61	-62	1	2	15	41	42	1	10	15	21	17	6	6	16	48	-46	2	3	17	49	50
1	5	14	61	-61	2	3	15	32	27	3	10	15	57	55	1	7	16	20	24	1	4	17	56	56
3	5	14	22	30	1	4	15	55	55	2	11	15	47	-48	3	7	16	21	-20	3	4	17	36	35
5	5	14	90	87	3	4	15	17	17	1	12	15	28	29	5	7	16	41	-41	4	5	17	52	53
0	6	14	20	-16	4	5	15	37	-40	0	0	16	54	-54	0	8	16	52	-51	2	7	17	64	-61
1	7	14	39	-40	3	6	15	34	-28	0	2	16	67	69	2	8	16	18	-22	1	8	17	69	-69
4	8	14	22	26	5	6	15	37	-37	2	2	16	26	-22	4	8	16	36	36	3	8	17	46	-46
3	9	14	63	63	2	7	15	37	32	0	4	16	81	79	6	8	16	35	33	1	1	18	49	-49
5	9	14	42	-42	1	8	15	74	-73	2	4	16	64	62	1	9	16	26	-17	1	3	18	34	-35
7	9	14	55	-58	3	8	15	30	-26	4	4	16	65	-66	5	9	16	18	15	3	3	18	44	43
9	9	14	57	57	5	8	15	54	55	3	5	16	21	22	0	10	16	74	75	2	4	18	20	-19
3	11	14	43	45	2	9	15	62	59	0	6	16	116	-115	2	10	16	20	-20	0	6	18	41	-40
7	11	14	61	-62	6	9	15	57	-56	2	6	16	22	22	4	10	16	62	-59	1	2	19	74	-71

Table A1.11 Structure Factors for Dehydrated Analcime

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	1	2	243	223	6	7	7	96	-91	1	1	10	134	-139	8	9	11	42	42	6	9	13	27	-23
0	2	2	120	-117	0	0	8	205	206	0	2	10	56	-46	1	10	11	25	-17	8	9	13	28	-17
1	2	3	91	-88	0	2	8	90	92	1	3	10	90	-95	3	10	11	36	-40	3	10	13	29	-34
2	3	3	217	215	2	2	8	46	-45	3	3	10	103	101	5	10	11	27	-6	5	10	13	26	-21
0	0	4	555	-526	1	3	8	105	110	0	4	10	67	73	7	10	11	50	44	7	10	13	46	44
0	2	4	40	-28	0	4	8	23	-14	2	4	10	28	31	2	11	11	45	43	2	11	13	44	-47
2	2	4	80	81	2	4	8	59	59	1	5	10	90	97	10	11	11	28	-28	2	13	13	50	-39
1	3	4	101	-100	4	4	8	54	-53	3	5	10	48	-49	0	2	12	66	-68	1	1	14	80	77
0	4	4	105	108	1	5	8	26	-17	5	5	10	112	-116	0	4	12	33	-36	1	3	14	59	55
4	4	4	35	27	3	5	8	52	-57	0	6	10	56	52	2	4	12	45	-48	3	3	14	45	-48
1	2	5	136	-140	0	6	8	136	-139	4	6	10	25	-29	4	4	12	33	35	0	4	14	28	-24
2	3	5	27	-16	2	6	8	40	44	1	7	10	31	30	0	6	12	115	116	2	4	14	23	1
1	4	5	40	-42	4	6	8	48	49	7	7	10	25	-8	2	6	12	29	-33	1	5	14	47	-46
3	4	5	33	25	6	6	8	76	-73	0	8	10	72	-74	4	6	12	71	-72	5	5	14	61	57
2	5	5	43	42	1	7	8	117	-122	4	8	10	41	29	6	6	12	58	55	1	7	14	24	-22
1	1	6	122	-121	3	7	8	47	41	6	8	10	30	30	1	7	12	33	31	7	7	14	30	17
0	2	6	70	68	5	7	8	81	81	1	9	10	27	-19	0	8	12	45	39	3	9	14	42	40
1	3	6	58	-55	8	8	8	41	41	3	9	10	41	-47	6	8	12	32	-25	5	9	14	43	-33
3	3	6	100	-102	1	2	9	119	120	5	9	10	65	68	8	8	12	33	-36	7	9	14	39	-39
0	4	6	173	-175	2	3	9	119	-123	7	9	10	54	57	0	10	12	47	-52	9	9	14	42	35
2	4	6	45	-48	1	4	9	29	31	9	9	10	72	-70	2	10	12	30	19	3	11	14	36	29
1	5	6	92	96	2	5	9	37	-34	0	10	10	46	-41	6	10	12	26	-27	7	11	14	37	-36
3	5	6	22	-21	4	5	9	34	27	1	2	11	54	-54	0	12	12	35	-37	4	12	14	28	-7
5	5	6	27	-28	1	6	9	100	-106	1	4	11	61	-68	8	12	12	47	44	1	2	15	39	34
0	6	6	127	-126	3	6	9	125	-126	2	5	11	27	21	1	2	13	30	-32	1	4	15	44	38
4	6	6	86	86	5	6	9	34	27	4	5	11	49	53	1	4	13	52	-48	4	5	15	32	-30
1	2	7	22	-17	2	7	9	42	48	1	6	11	23	30	3	4	13	26	-21	1	8	15	46	-47
2	3	7	150	-153	4	7	9	42	39	2	7	11	43	-44	4	5	13	44	-45	5	8	15	38	38
1	4	7	115	120	6	7	9	57	-64	1	8	11	84	85	1	6	13	28	33	2	9	15	36	37
3	4	7	41	47	1	8	9	37	-34	3	8	11	31	29	3	6	13	34	38	6	9	15	36	-32
4	5	7	67	-68	5	8	9	28	34	5	8	11	72	-69	1	8	13	43	45	3	10	15	39	33
1	6	7	34	26	7	8	9	41	-44	7	8	11	24	3	3	8	13	26	17	2	11	15	33	-35
3	6	7	85	88	2	9	9	66	-68	2	9	11	32	-28	5	8	13	42	-45	0	0	16	62	-51
2	7	7	179	179	6	9	9	32	41	4	9	11	28	-32	2	9	13	50	45	0	2	16	55	46

Table A1.11 Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	4	16	48	50	4	6	16	61	55	0	10	16	52	46	2	7	17	46	-35	1	3	18	36	-26
2	4	16	41	34	6	6	16	35	-30	2	3	17	31	33	1	8	17	50	-42	3	3	18	40	31
4	4	16	43	-41	0	8	16	45	-42	1	4	17	38	39	3	8	17	30	-27	1	2	19	44	-41
0	6	16	83	-77	1	9	16	36	-13	4	5	17	39	36										

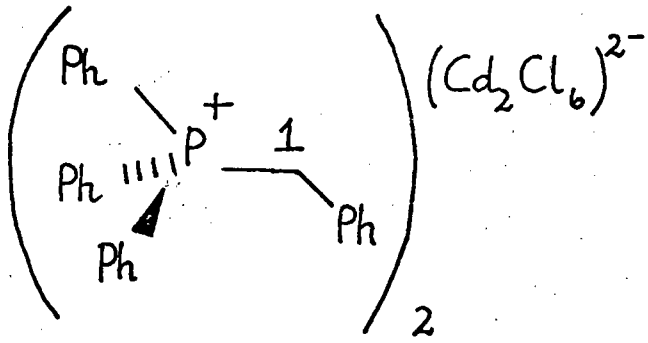
APPENDIX TWO

EXPLANATION OF TERMS

Each compound is identified by the CCDC code. All distances are in Å and angles in degrees.

d	=	bond length or interatomic distance
dv	=	sum of the van der Waals radii (refer to Table 13.1)
Δd	=	corrected interatomic distance (d - dv)
Cj – P – Ci etc	=	bond angles associated with the phosphonium ions
Ci – P ---X etc	=	the angles between the approaching nucleophile X^- and the groups attached to the phosphorus atom
ϕ	=	the angle as defined in Figure 13.2 and calculated as shown in Appendix 3. In the case of P^+--- X^- distances ϕ refers to the closest X^- groups to the phosphorus atoms.
C_α ---- X^-	=	the distances between the nucleophile X^- and the α-carbon atoms of ligands on the phosphorus atoms.

1. BPPNCD[122]: BIS(BENZYLTRIPHENYLPHOSPHONIUM) HEXACHLORO-DI-CADMIUM



a = 20.593, b = 12.832,
c = 12.730Å, α = 120.84,
β = 117.84, γ = 83.97°.
PĪ Z = 2

angles Cj-P-Ci

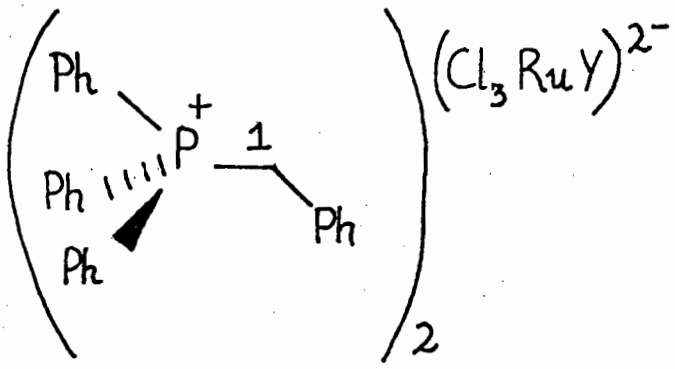
	i (P1)				i (P2)			
	1	2	3	4	1	2	3	4
1		107.9	105.7	113.3		107.2	108.4	110.6
2			111.0	112.4			110.8	108.3
j 3				106.3	j 3			111.6
ϕ			118.7	235.0	ϕ		119.6	242.2

bond lengths P-Ci

P1				P2			
1.805	1.770	1.782	1.755	1.797	1.778	1.784	1.782

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P1----Cl	4.44	3.55	0.89	53.6	81.4	73.2	164.5
P1----Cl	4.66	3.55	1.02	49.2	156.3	84.5	78.1
P2----Cl	4.39	3.55	0.84	55.7	81.4	73.1	165.8
P2----Cl	4.78	3.55	1.23	40.7	88.2	148.8	83.7
Cα(P1)----Cl	3.67	3.45	0.22				
Cα(P2)----Cl	3.69	3.45	0.24				
ϕ(P1) = 295.7°							
ϕ(P2) = 293.6°							

2. BZPHRU[123]: BENZYLTRIPHENYLPHOSPHONIUM BICYCLO(2.2.1)HEPTA-2,5-DIENE-(CARBONYL)-TRICHLORO-RUTHENIUM(II)



a = 12.471, b = 18.176,
c = 14.181 Å, β = 109.84°
$P2_1/c$ Z = 4

angles Cj – P – Ci

		i		
	1	2	3	4
1		109.8	111.5	108.3
2			108.9	111.3
j 3				107.0
ϕ			120.7	238.4

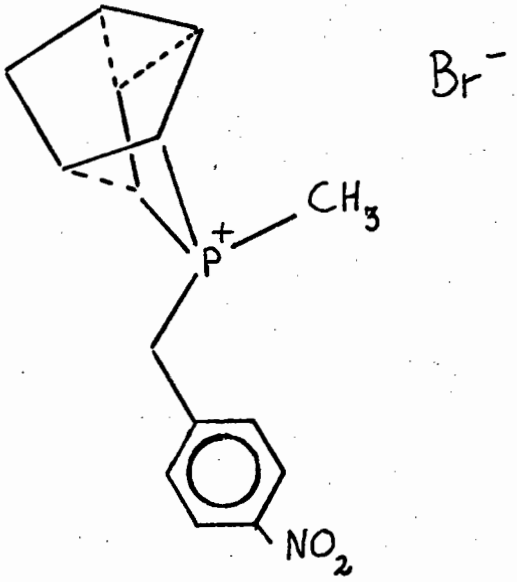
bond lengths P – Ci

1.818	1.797	1.800	1.793

Secondary bonds	d	dv	Δd	angles	C_i – P------X		
P ---- Cl	4.63	3.55	1.08	50.9	75.6	161.1	87.5
P ---- Cl!	4.94	3.55	1.39	39.2	142.3	77.7	101.3
Cα ----Cl	4.15	3.45	0.70				

ϕ = 309.3°

3. MPTCOB[124]: 4-METHYL-4-*p*-NITROBENZYL-4-PHOSPHONIOTETRACYCLO(3.3.0-2,8-.0-3,6-)OCTANE BROMIDE



a = 6.750, b = 8.705
c = 12.961Å α = 91.60,
β = 95.02, γ = 89.27°
$P\bar{1}$ Z = 2

angles Cj — P — Ci

	i			
	1	2	3	4
1		118.2	116.7	107.0
2			74.8	119.0
j 3				119.0
ϕ			86.3	222.5

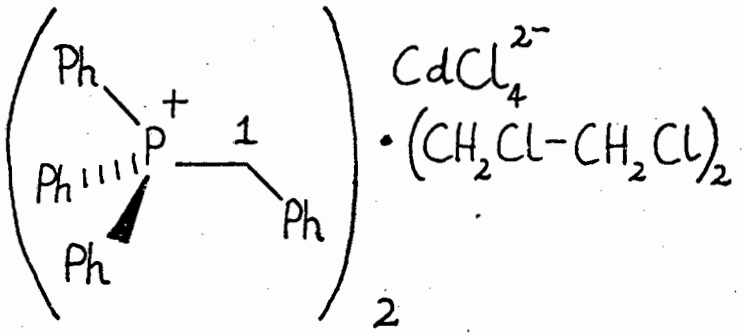
bond lengths P — Ci

1.813	1.815	1.822	1.768

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	3.73	3.65	0.08	77.0	78.1	152.9	74.3
Cα---- Br	3.70	3.55	0.15				

ϕ = 291.4°

4. PBPCCD[125]: BIS(TRIPHENYLBENZYLPHOSPHONIUM) TETRACHLOROCADMIUM DICHLORO-ETHANE CLATHRATE



a = 14.73, b = 24.88,
c = 16.37Å, β = 112.61°,
$P2_1/c$ Z = 4

angles Cj — P — Ci

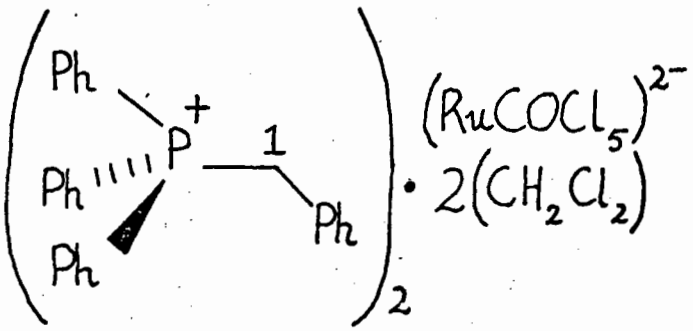
	i (P1) 1	2	3	4		i (P2) 1	2	3	4
1		112.6	111.5	106.7	1		112.2	112.4	108.1
2			106.5	111.3	2			106.9	109.6
j 3				108.1	j 3				107.6
ϕ			119.6	237.7	ϕ			120.4	239.1

bond lengths P — Ci

P1				P2			
1.802	1.806	1.791	1.828	1.779	1.755	1.782	1.759

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P1 ---- Cl	4.97	3.55	1.42	43.8	84.5	89.8	150.4
P1 ---- Cl'	4.45	3.55	0.90	53.8	165.7	79.9	77.7
P2 ---- Cl	4.90	3.55	1.35	42.9	74.0	145.1	104.5
P2 ---- Cl''	4.53	3.55	0.98	61.6	169.8	70.4	80.5
P2 ---- Cl	4.99	3.55	1.44	175.0	63.0	69.0	75.5
Cα(P1) ---- Cl	4.00	3.45	0.55				
Cα(P2) ---- Cl	3.66	3.45	0.21				
ϕ(P1) = 185.0°							
ϕ(P2) = 188.9°							

5. BZPPRU[126]: BENZYLTRIPHENYLPHOSPHONIUM CARBONYL-PENTACHLORO-RUTHENIUM(III) DICHLOROMETHANE SOLVATE



a = 13.913, b = 18.401, c = 20.965Å, *Pbcn* Z = 4

angles Cj — P — Ci

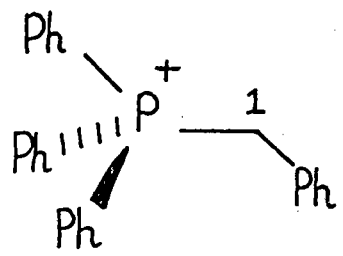
	i 1	2	3	4
j 1		108.6	112.7	107.4
2			109.7	110.7
3				107.8
ϕ			121.7	240.3

bond lengths P ---- Ci

1.823	1.802	1.800	1.833

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Cl	4.95	3.55	1.40	38.0	132.4	114.9	70.9
P ---- Cl	4.39	3.55	0.84	55.3	161.3	73.1	85.1
P ---- Cl	4.68	3.55	1.13	161.0	54.2	83.1	75.6
Cα --- Cl	3.69	3.45	0.24				
ϕ = 190.6°							

6. PBPBSN[127]: BENZYL-TRIPHENYLPHOSPHONIUM DICHLOROTRIBUTYL-TIN



$[SnCl_2(C_4H_9)_2]^-$

a = 9.8521
b = 16.9142 Å
c = 22.3517 Å
β = 91.4235°
$P2_1/c$
Z = 4

angles Cj – P – Ci

	i 1	2	3	4
1		108.4	112.6	106.0
2			109.3	110.4
j 3				109.9
ϕ			121.0	241.5

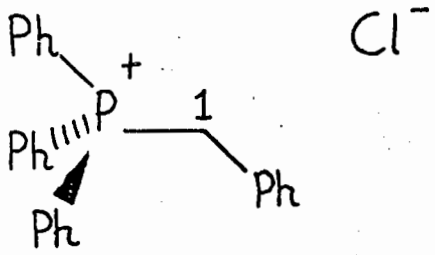
bond lengths P – Ci

1.808	1.806	1.788	1.827

Secondary bonds	d	dv	Δd	angles	Ci --- P ---- X		
P ---- Cl	4.69	3.55	1.14	46.1	154.4	83.7	84.1
Cα ---- Cl	3.67	3.45	0.22				

ϕ = 182.6°

7. BZPPOS[128]: BENZYLTRIPHENYLPHOSPHONIUM CHLORIDE



a = 17.147 Å
b = 18.147 Å
c = 13.860 Å
Pbca
Z = 8

angles Cj — P — Ci

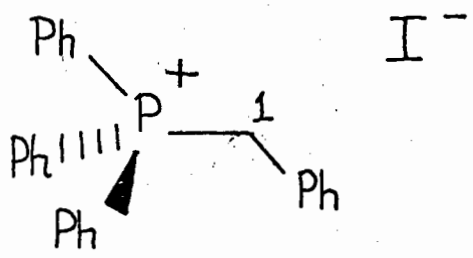
		j		
	1	2	3	4
1		111.7	108.8	108.9
i 2			110.1	108.0
3				109.2
ϕ_i			121.8	240.8

bond lengths P — Ci

1.811	1.795	1.785	1.777

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Cl	4.30	3.55	0.75	55.7	165.3	82.8	72.8
P ---- Cl	4.42	3.55	0.87	48.6	80.4	157.2	85.2
Cα---- Cl	3.56	3.45	0.11				
ϕ = 306.0°							

8. [118]: BENZYLTRIPHENYLPHOSPHONIUM IODIDE



a = 9.692 Å
b = 21.580Å
c = 11.211Å
β = 107.2°
$P2_1/c$
Z = 4

angles Cj — P — Ci

	i 1	2	3	4
1		109.2	109.1	110.4
2			109.4	110.7
j 3				108.0
ϕ			119.5	238.1

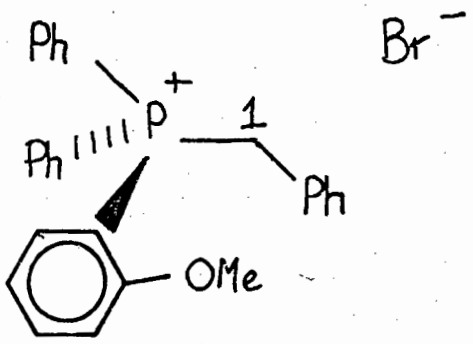
bond lengths P — Ci

1.808	1.780	1.800	1.801

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- I	5.01	3.78	1.23	43.8	152.2	89.8	81.0
P ---- I	4.64	3.78	0.86	56.8	73.0	81.5	167.3
Cα---- I	3.96	3.68	0.28				

ϕ = 78.4°

9. BMPPHB[129]: BENZYL(2-METHOXYPHENYL)DIPHENYLPHOSPHONIUM BROMIDE



a = 15.528 Å
b = 16.892 Å
c = 17.921 Å
Pbca
Z = 8

angles Cj — P — Ci

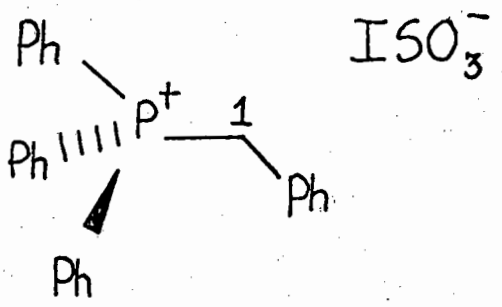
j \ i	1	2	3	4
1		108.3	109.0	107.9
2			109.5	110.1
3				111.9
ϕ			119.1	240.8

bond lengths P — Ci

1.815	1.799	1.840	1.823

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.65	3.65	1.00	51.0	80.0	80.3	158.9
P ---- Br	4.59	3.65	0.94	48.1	85.4	156.8	77.4
P ---- O	2.88	3.30	-0.42	161.11	53.5	76.6	85.5
Cα---- Br	3.64	3.55	0.09				
ϕ = 294.2°							

10. BZTPPI[130]: BENZYLTRIPHENYLPHOSPHONIUM IODOSULPHINATE



a = 10.92 Å
b = 17.643Å
c = 15.49 Å
β = 125.9
$P2_1/c$
Z = 4

angles Cj — P —— Ci

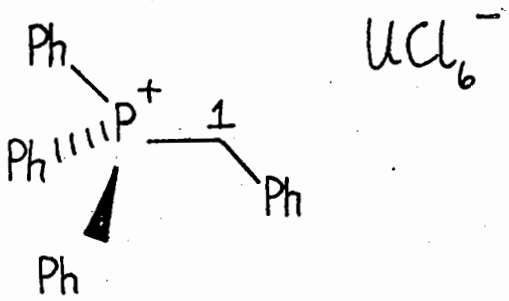
j \ i	1	2	3	4
1		104.7	113.1	110.3
2			109.0	111.3
3				108.5
ϕ			118.5	240.3

bond lengths P — Ci

1.813	1.796	1.783	1.795

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- O	4.81	3.30	1.51	92.8	160.1	54.2	70.1
P ---- O	4.74	3.30	1.44	169.6	82.9	70.0	59.7
P ---- I	4.94	3.78	1.16	61.8	78.5	172.2	69.7
Cα---- I	4.39	3.68	0.71				
ϕ = 292.0°							

11. TPBZPU[131]: BENZYLTRIPHENYLPHOSPHONIUM HEXACHLOROURONATE



a = 11.72 Å
b = 18.78 Å
c = 13.61 Å
β = 105.71°
$P2_1/c$
Z = 4

angles Cj — P — Ci

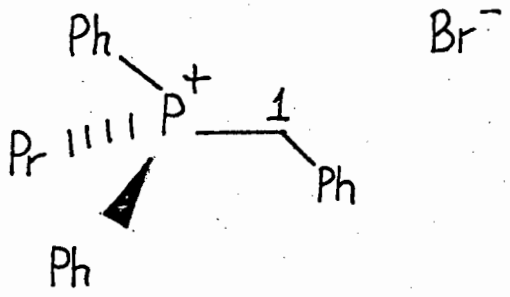
j \ i	1	2	3	4
1		108	111	110
2			110	109
3				109
ϕ			121	241

bond lengths P — Ci

1:80	1:78	1:80	1:80

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Cl	4.80	3.55	1.25	168	81	57	74
P ---- Cl	4.81	3.55	1.26	53	71	89	160
P ---- Cl	4.88	3.55	1.33	73	64	174	74
Cα---- Cl	4.00	3.45	0.55				
ϕ = 138°							

12. MPBPBR[132]: BENZYLMETHYLPHENYLPROPYL PHOSPHONIUM BROMIDE



a = 12.85 Å
b = 8.97 Å
c = 14.66 Å
$P2_12_12_1$
Z = 4

angles Cj – P – Ci

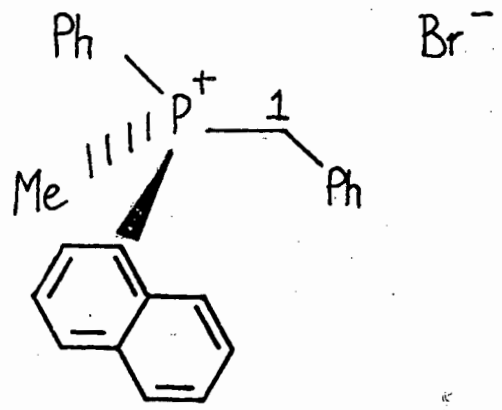
j \ i	1	2	3	4
1		112.4	103.3	112.2
2			110.7	110.8
3				107.2
ϕ			119.4	234.3

bond lengths P – Ci

1.818	1.757	1.812	1.752

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.22	3.65	0.57	68.1	178.5	67.8	70.1
P ---- Br	4.62	3.65	0.97	57.6	83.6	68.3	165.4
Cα---- Br	3.92	3.55	0.37				
ϕ = 178.5°							

13. BZMNPB[133]: BENZYLMETHYL(1-NAPHTYL)PHENYLPHOSPHONIUM BROMIDE



a = 22.12 Å
b = 9.89 Å
c = 9.49 Å
$P2_12_12_1$
Z = 4

angles Cj — P — Ci

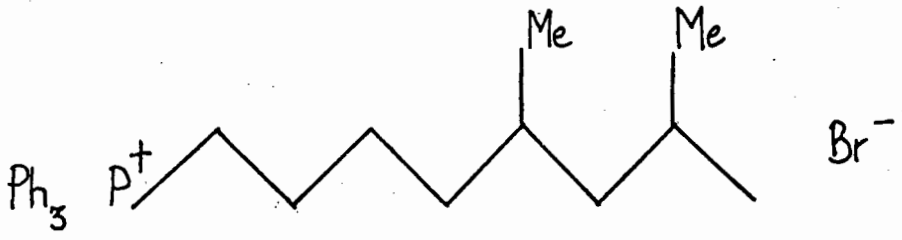
	i			
	1	2	3	4
1		112.8	106.4	109.6
2			109.9	110.4
j 3				107.6
ϕ			120.6	236.6

bond lengths P — Ci

1.823	1.789	1.778	1.802

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.70	3.65	1.05	148.8	83.7	42.5	87.0
P ---- Br	4.25	3.65	0.60	68.4	170.7	61.6	76.9
Cα---- Br	3.96	3.55	0.41				
ϕ = 170.0°							

14. MOCTPB[134]: (3,7-DIMETHYL-2,6-OCTADIENYL)TRIPHENYLPHOSPHONIUM BROMIDE



a = 12.614 Å
b = 13.845 Å
d = 15.838 Å
β = 113.35°
$P2_1/c$
Z = 4

angles Cj — P — Ci

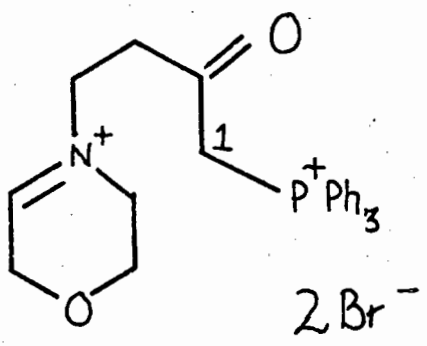
		i		
	1	2	3	4
1		109.1	110.2	110.4
j 2			110.4	108.9
3				107.8
ϕ			121.4	240.4

bond lengths P — Ci

1.805	1.790	1.794	1.790

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.67	3.65	1.02	46.4	152.0	74.6	94.8
P ---- Br	4.44	3.65	0.79	57.6	80.7	75.2	167.2
Cα---- Br	3.66	3.55	0.11				
ϕ = 65.0°							

15. OMBPPB[135]: (2-OXO-4-MORPHOLINO)-BUTYL-TRIPHENYLPHOSPHIUM DIBROMIDE



a = 12.736 Å
b = 12.792 Å
c = 9.663 Å
α = 115.80°
β = 105.12°
γ = 66.53°
$P\bar{1}$
Z = 2

angles Cj — P — Ci

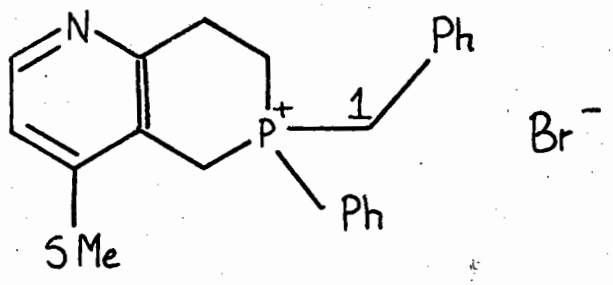
j \ i	1	2	3	4
1		111.0	111.3	105.8
2			109.8	108.2
3				110.5
ϕ			122.6	242.8

bond lengths P — Ci

1.812	1.806	1.807	1.778

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.69	3.65	1.04	45.5	135.4	89.1	77.8
P ---- Br	4.34	3.65	0.71	58.0	68.9	166.2	83.3
Cα---- Br	3.66	3.55	0.11				
ϕ = 314.1°							

16. HTPOPY[136]: (-)-(5)-6-BENZYL-5,6,7,8-TETRAHYDRO-4-(METHYLTHIO)-6-PHENYL-PHOSPHONIO-(4,3-D)-PYRIMIDINIUM BROMIDE



a = 11.087 Å
b = 8.763 Å
c = 12.181 Å
β = 116.38°
$P2_1$
Z = 2

angles Cj — P — Ci

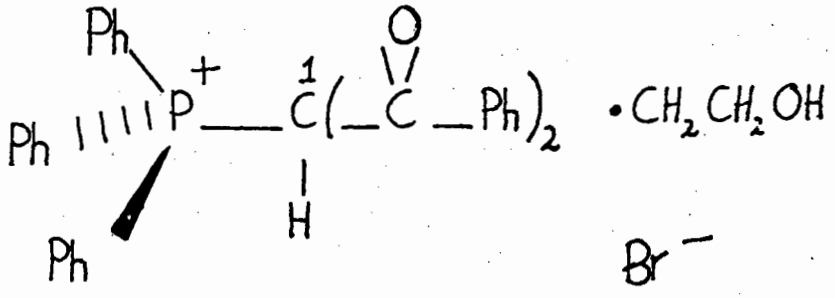
j \ i	1	2	3	4
1		111.6	111.5	110.5
2			108.6	111.6
3				102.6
ϕ			121.6	235.2

bond lengths P — Ci

1.779	1.810	1.771	1.787

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.20	3.65	0.55	171.8	76.3	66.4	63.3
P ---- Br	4.15	3.65	0.50	89.5	64.0	62.6	159.0
Cα---- Br	4.50	3.55	0.95				
ϕ = 61.6°							

17. TPBOME[137] : TRIPHENYLPHOSPHONIA-DIBENZOYLMETHANE BROMIDE ETHANOL SOLVATE



a = 8.727 Å
b = 17.922Å
c = 19.060Å
cc
Z = 4

angles Cj — P — Ci

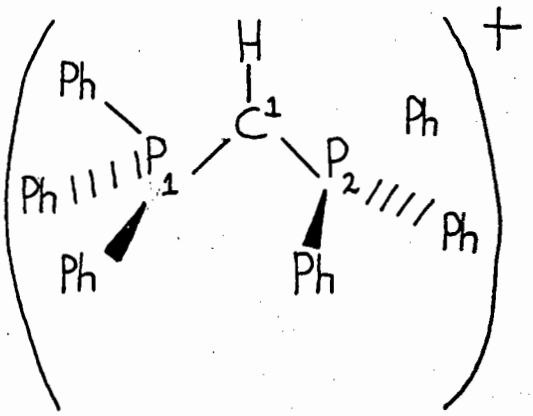
j \ i	1	2	3	4
1		105.7	107.2	116.0
2			110.2	105.9
3				107.2
ϕ			117.5	243.1

bond lengths P — Ci

1.842	1.751	1.803	1.811

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- Br	4.45	3.65	0.80	57.6	70.7	78.4	169.9
Cα---- Br	4.20	3.55	0.65				
P ---- O(1)	2.94	3.32	-0.38				
P ---- O(2)	3.32	3.32	0				
ϕ = 305.8°							

18. TPMYPBIO[138]: ((TRIPHENYLPHOSPHORANYLIDENE)METHYL) TRIPHENYLPHOSPHONIUM BROMIDE



a = 9.666 Å
b = 19.187Å
c = 17.116Å
β = 102.675°
$P2/c$
Z = 4

angles Cj — P — Ci

	i P(1)					i (P2)			
	1	2	3	4		1	2	3	4
1		112.4	116.0	109.3	1		112.1	112.8	112.5
j 2			108.1	106.0	j 2			105.3	105.8
3				104.3	3				107.8
ϕ			125.0	242.7	ϕ			118.6	241.0

bond lengths P(1) — Ci

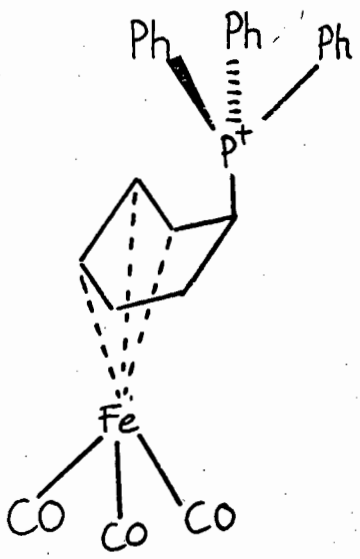
1.710	1.808	1.810	1.806

bond lengths P(2) — Ci

1.695	1.818	1.806	1.802

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X	
P(2)----Br	4.82	3.65	1.17	49.7	89.8	
Cα ----Br	3.94	3.55	0.39		78.0	160.7
ϕ = 290.5°						

19. TPHDFC[139]: (5-EXO-TRIPHENYLPHOSPHONIUM-CYCLOHEXA-1,3-DIENE) TRICARBONYL-IRON TETRAFLUORABORATE



BF_4^-

a = 9.24 Å
b = 18.85 Å
c = 16.82 Å
β = 118.04°
$P2_1/c$
Z = 4

angles Cj — P — Ci

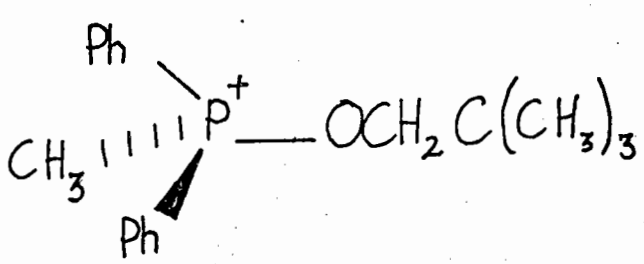
	i 1	2	3	4
1		108.6	110.6	108.2
j 2			109.4	112.5
3				107.7
ϕ			120.0	237.7

bond lengths P — Ci

1.825	1.783	1.806	1.813

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- F	4.33	3.27	1.06	44.8	150.3	77.2	91.7
ϕ = 195.8°							

20. DPHNPP[140]: NEOPENTYLOXY-(METHYL)-(DIPHENYL)-PHOSPHONIUM BROMIDE



Br^-

a = 9.148 Å
b = 10.451Å
c = 19.250Å
β = 92.02°
$P2_1/c$
Z = 4

angles Cj — P — Ci/Oi

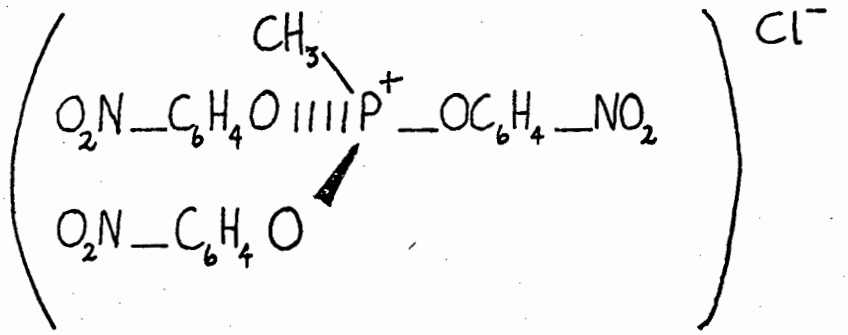
	i 1	2	3	4
1		109.8	104.2	111.9
2			111.9	110.4
j 3				108.7
ϕ			119.9	237.1

bond lengths P — Ci/Oi

1.568	1.785	1.790	1.779

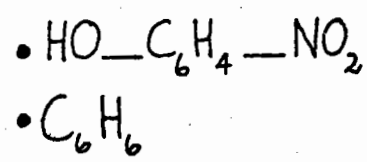
Secondary bonds	d	dv	Δd	angles	Ci/Oi — P ---- X		
P ---- Br	4.611	3.65	0.96	162.3	79.5	85.5	50.4
ϕ = 241.1°							

21. MNPXPH[141]: METHYL-TRI(p-NITROPHENOXY)-PHOSPHONIUM CHLORIDE p-NITROPHENOL BENZENE SOLVATE



Cl^-

a = 11.940 Å
b = 12.600 Å
c = 10.471 Å
α = 108.26°
β = 95.42°
γ = 84.58°
$P\bar{1}$
Z = 2



angles Oj – P – Oi/Ci

	i 1(O)	2(O)	3(O)	4(C)
1		103.9	105.6	113.4
2			110.3	116,7
j 3				106,5
ϕ			116.1	232,4

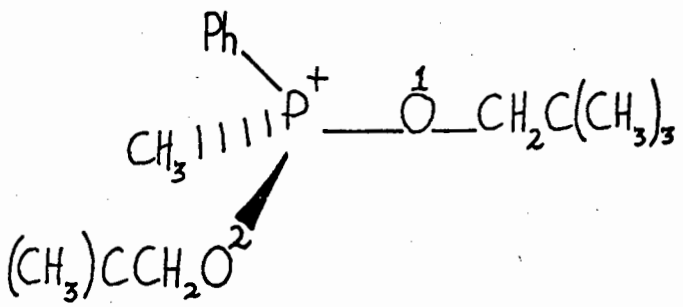
bond lengths P – Ci/Oi

1.553	1.552	1.549	1.717

Secondary bonds	d	dv	Δd	angles	Ci/Oi – P ---- X		
P ---- Cl	4.081	3.55	0.53	68.8	162.3	87.3	56.4
P ---- Cl	4.356	3,55	0,80	88.8	83.9	155.9	49.5

ϕ = 196.9°

22. MPXPBR[140]: DI(NEOPENTYLOXY)-METHYL-PHENYL-PHOSPHONIUM BROMIDE



a = 13.621 Å
b = 12.002 Å
c = 12.502 Å
β = 102.27°
$P2_1/n$
Z = 4

angles Cj/Oj — P — Ci/Oi

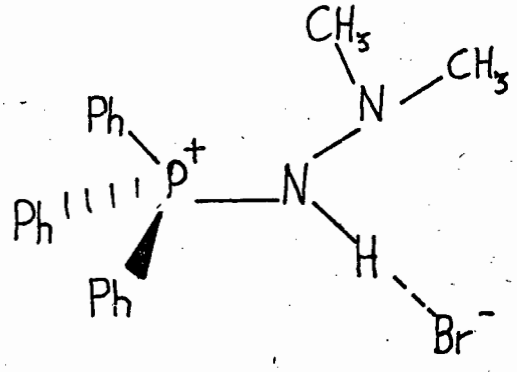
	1(O)	i 2(O)	3	4
1		111.4	103.3	111.0
2			113.5	103.9
j 3				114.0
ϕ			122.1	244.8

bond lengths P — Ci/Oi

1.545	1.552	1.757	1.771

Secondary bonds	d	dv	Δd	angles	Ci/Oi — P ---- X		
P ---- Br	4.43	3.65	0.78	69.5	166.4	54.3	88.0
P ---- Br	4.99	3.65	1.34	113.6	65.8	48.5	134.9
ϕ = 194.6°							

23. MHZPPB[142]: N',N'-DIMETHYLHYDRAZINO-TRIPHENYLPHOSPHONIUM BROMIDE



a = 8.42 Å
b = 17.93 Å
c = 16.47 Å
β = 127.4°
$P2_1/c$
Z = 4

angles Cj — P — Ci/N

	1(N)	i 2	3	4
1		110.1	106.0	113.4
2			109.9	109.5
j 3				107.9
ϕ			118.7	237.0

bond lengths P — Ci/N

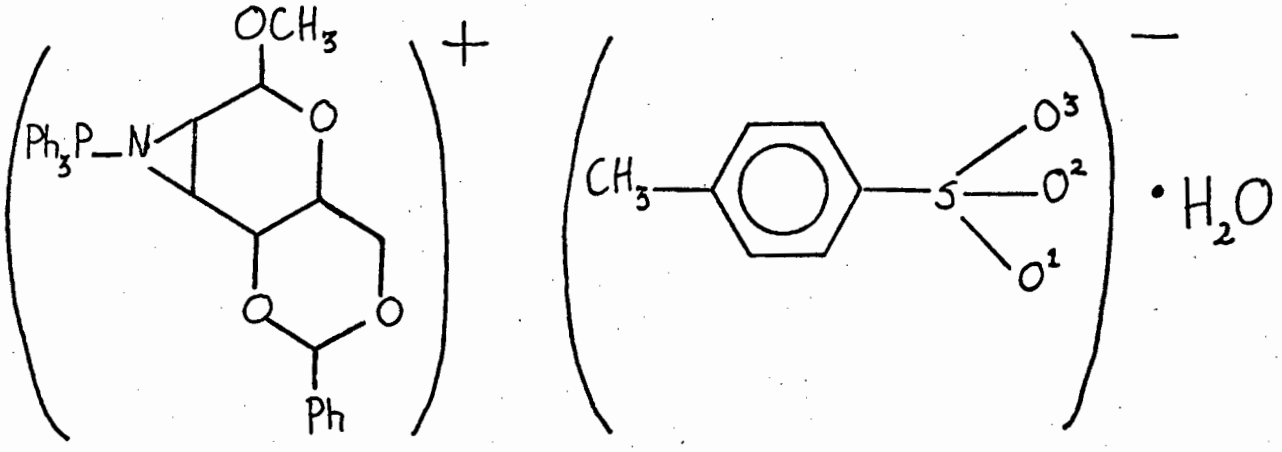
1.641	1.786	1.775	1.778

Secondary bonds	d	dv	Δd	angles	Ci/N — P ---- X		
P ---- Br	4.22	3.65	0.57	45.5	152.3	73.3	94.8
ϕ = 195.7°							

Hydrogen bonding

d(N — H)	0.86
d(N --- Br⁻)	3.28
∢(N — H --- Br)	175.1°

24. MBYPAT[143]: METHYL-4,6-O-BENZYLIDENE-2,3-DIDEOXY-2,3-(N-TRIPHENYL-PHOSPHONIO-EPIMINO)-ALPHA-D-ALLOSIDE P-TOLUENESULFONATE MONOHYDRATE



a = 17.849 Å, b = 15.414 Å, c = 13.338 Å, $P2_12_12_1$, Z = 4

angles Cj — P — Ci/N

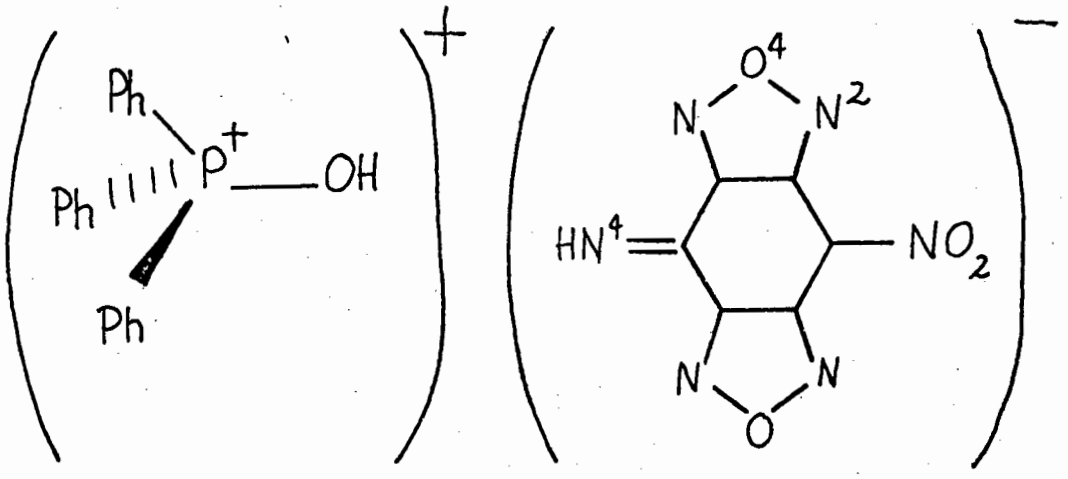
		i		
	1(N)	2	3	4
1		106.7	110.3	109.9
2			111.4	109.0
j 3				109.4
ϕ			121.1	242.0

bond lengths P — Ci/N

1.677	1.792	1.787	1.803

Secondary bonds	d	dv	Δd	angles	Ci/N — P ---- X		
P ---- O(2)	4.15	3.22	0.83	168.5	76.8	58.6	78.5
ϕ = 105.6 °							

25. TPONBZ[144]: HYDROXY-TRIPHENYLPHOSPHONIUM 4-IMINO-8-ACI-NITROBENZO(1,2-C.4,5-C')DIFURAZAN



a = 9.615 Å, b = 10.828 Å, c = 11.226 Å, α = 81.82°, β = 97.72°, γ = 99.67°, $P\bar{1}$, Z = 2

angles Cj — P — Ci/O

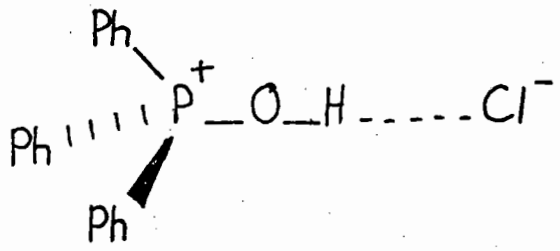
		i 1(O)	2	3	4
	1		115.6	108.6	107.5
	2			107.2	107.5
j	3				110.4
	ϕ			120.4	240.0

bond lengths P — Ci/O

1.639	1.767	1.793	1.794

Secondary bonds	d	dv	Δd	angles	Ci/O — P ---- X		
P ---- N(4)	4.83	3.35	1.48	89.0	48.3	79.7	155.6
P ---- N(2)	3.88	3.35	0.53	53.4	97.9	154.3	65.7
P ---- O(4)	4.22	3.32	0.90	73.0	90.7	158.2	50.6
O(1)---N(2)	3.190 (H bond)						
ϕ = 279.2°							

26. HXTPPC[145]: HYDROXO-TRIPHENYLPHOSPHONIUM CHLORIDE



a = 9.788 Å
b = 16.477Å
c = 10.568Å
β = 112.11°
$P2_1/n$
Z = 4

angles Cj — P — Ci/O

	i 1(O)	2	3	4
1		111.7	107.2	107.2
2			109.3	108.9
j 3				108.0
ϕ			119.6	240.9

bond lengths P — Ci/O

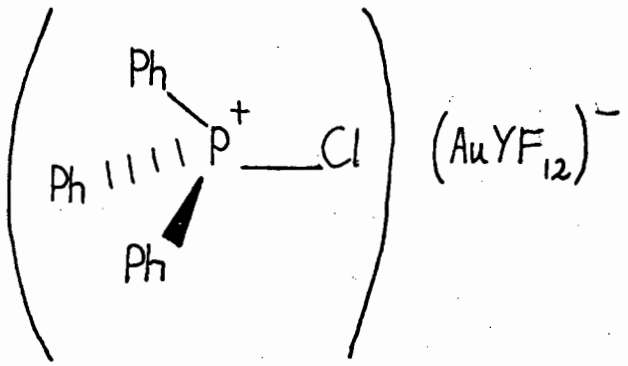
1.512	1.784	1.779	1.782

Secondary bonds	d	dv	Δd	angles	Ci/O — P ---- X		
P ---- Cl	3.86	3.55	0.31	34.5	83.5	138.0	104.8
ϕ = 322.6°							

Hydrogen bonding

d(O — H)	1.12
d(O --- Cl)	2.75
<(O — H --- Cl)	176.6

27. CPPSAN[146]: CHLOROTRIPHENYLPHOSPHONIUM BIS(CIS-1,2-BIS(TRIFLUOROMETHYL)-ETHENE-1,2-DITHIOLATO) GOLD



a = 13.166 Å
b = 11.623 Å
c = 20.486 Å
β = 91.21°
$P2_1/c$
Z = 4

angles Cj — P — Ci/Cl

	i 1(Cl)	2	3	4
1		109.3	107.6	106.7
2			111.5	111.7
j 3				109.7
ϕ			121.2	239.1

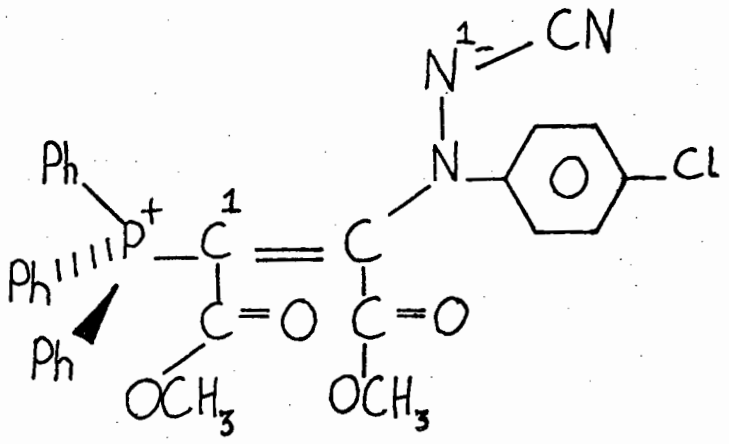
bond lengths P — Ci/Cl

1.982	1.755	1.764	1.762

Secondary bonds	d	dv	Δd	angles	Ci/Cl — P ---- X		
P ---- F	4.85	3.27	1.58	92.1	82.0	41.2	150.2
P ---- F	4.54	3.27	1.27	63.5	74.6	73.5	170.1

ϕ = 299.3°

28. TPEAZMIO[147]: N-2--(P-CHLOROPHENYL)-N-1--CYANO-2--(1,2-BIS(METHOXY-CARBONYL)-2-(TRIPHENYL-PHOSPHONIO)VINYL) HYDRAZIDE



a = 11.868 Å
b = 12.584 Å
c = 11.682 Å
α = 110.20°
β = 117.7°
γ = 68.55°
$P\bar{1}$
Z = 2

angles Cj — P — Ci

	i 1	2	3	4
1		115.0	109.1	107.9
2			113.1	108.3
j 3				102.7
ϕ			128.2	239.1

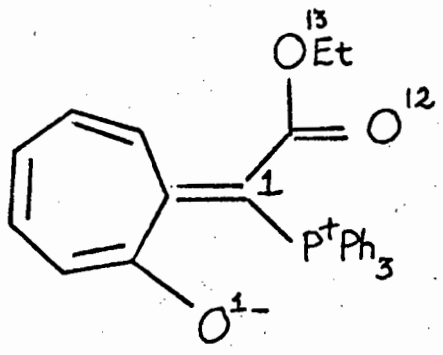
bond lengths P — Ci

1.753	1.796	1.807	1.825

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- O(33)	4.510	3.32	1.19	31.5	143.7	81.8	99.6
P ---- N(21)	3.200	3.35	0.15	48.3	104.4	71.6	146.2
P ---- N(36)	2.88	3.35	-0.67	68.2	80.3	72.3	171.4

ϕ = 292.8°

29. TROPBT[148]: 2-TROPONYL-(ETHOXYCARBONYL)-METHYLENE-TRIPHENYLPHOSPHONIUM)-BETAINE



a = 16.94 Å
b = 8.79 Å
c = 15.71 Å
$Pna2_1$
Z = 4

angles Cj — P — Ci

	i 1	2	3	4
1		121.1	120.4	100.5
2			111.5	102.8
j 3				93.4
ϕ			147.9	248.1

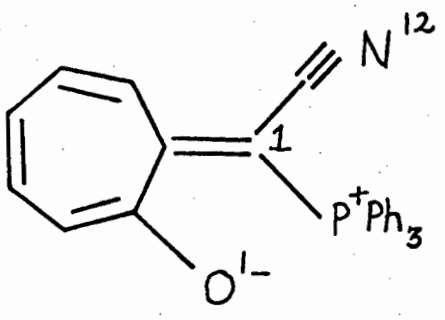
bond lengths P — Ci

1.76	1.82	1.84	1.86

Secondary bonds	d	dv	Δd	angles	Ci — P — X		
P --- O(1)	2.14	3.32	-1.18	79.9	83.0	80.4	172.8
P --- O(13)	3.05	3.32	-0.27	49.9	94.7	149.8	65.2
P --- O(12)	3.86	3.32	0.54	26.6	127.7	120.9	73.9

ϕ = 284.6°

30. TRCMPP[149]: 2-TROPONYLCYANOMETHYLENETRIPHENYLPHOSPHONIUM BETAINE



a = 9,68 Å
b = 16.14Å
c = 9,95 Å
α = 52.62°
β = 108.15°
γ = 116.78°
$P\bar{1}$
Z = 2

angles Cj — P — Ci

		i		
	1	2	3	4
1		112,3	103.0	118.6
2			104.6	115.9
j 3				99.5
ϕ			111.9	220.6

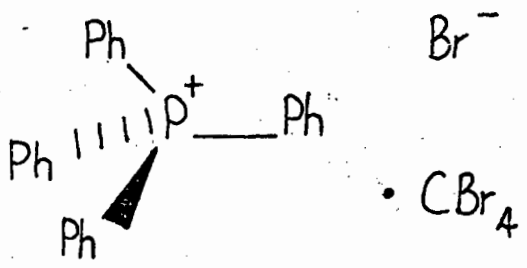
bond lengths P — Ci

1.70	1.77	1.83	1.78

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P --- O(1)	2.36	3.32	-0.96	74.9	77.0	177.8	81.2
P --- N(12)	3.57	3.35	0.22	38,6	120.7	64.4	123.3
P --- O(32)	4,60	3,32	1.28	102.4	68.4	154.4	65.0

ϕ = 291.3°

31. TPCBBR[150]: TETRAPHENYLPHOSPHONIUM CARBON TETRABROMIDE BROMIDE



a = 20.19 Å
b = 15.36 Å
c = 8.630 Å
β = 93.20°
$P2_1/a$
Z = 4

angles Cj — P — Ci

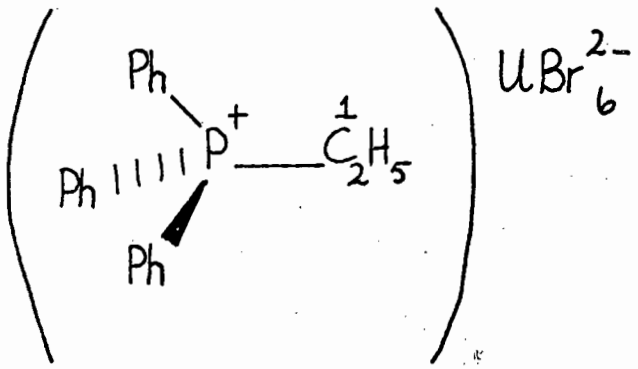
	i 1	2	3	4
1		104.3	112.9	110.7
2			110.8	110.4
j 3				107.7
ϕ			120.3	241.3

bond lengths P — Ci

1.829	1.811	1.805	1.786

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- BR	5.76	3.65	2.11	67.7	159.3	57.2	89.7
P ---- BR	6.60	3.65	2.95	99.5	47.8	146.4	70.1
P ---- BR	6.56	3.65	2.91	48.3	53.9	63.0	144.7

32. TPEPUB[151]: BIS(TRIPHENYLETHYLPHOSPHONIUM) HEXABROMO-URANIUM(IV)



a = 10.45 Å
b = 13.51 Å
c = 15.46 Å
β = 96.67°
$P2_1/n$
Z = 2

angles (°) Cj – P – Ci

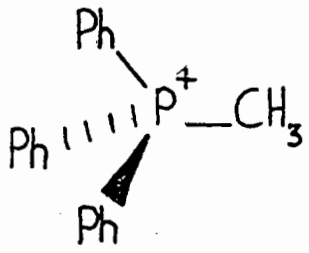
	i 1	2	3	4
1		106.3	109.6	109,3
2			110,2	110.6
j 3				110,8
ϕ			119,0	240.7

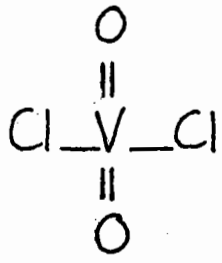
bond lengths P – Ci (Å)

1.783	1.814	1.775	1.785

Secondary bonds	d(Å)	dv	Δd	angles	Ci — P ---- X		
P ---- BR	4.72	3,65	1,07	159,2	52.9	81.5	81,5

33. PHCLOV[152]: METHYL-TRIPHENYLPHOSPHONIUM DICHLORODIOXO-VANADIUM





a = 10.359 Å
b = 12.772 Å
c = 14.697 Å
$P2_12_12_1$
Z = 4

angles Cj — P — Ci

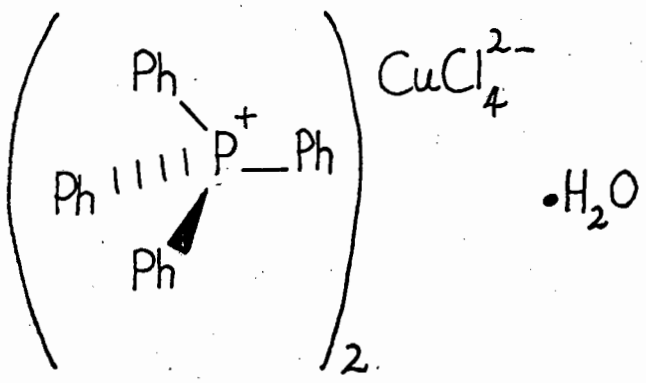
	j 1	2	3	4
1		110.0	110.0	108.4
2			109.8	109.3
i 3				109.3
ϕ			121.0	240.6

bond lengths P — Ci

1.793	1.798	1.791	1.769

Secondary bonds	d	dv	Δd	angles	Ci —— P ---- X		
P ---- Cl	7.12	3.55	3.57	40.6	141.2	73.5	105.3
P ---- Cl	7.40	3.55	3.85	105.3	137.5	78.5	35.0

34. TPHTCC[153]: BIS(TETRAPHENYLPHOSPHONIUM) TETRACHLOROCOPPER(II) MONOHYDRATE



a = 20.059 Å
b = 13.544 Å
c = 16.934 Å
β = 108.93°
$P2_1/c$
Z = 4

angles Cj – P – Ci

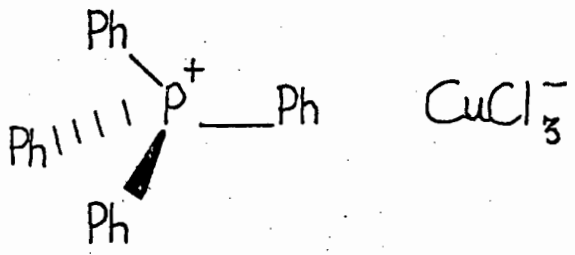
	P1					P2			
		i					i		
	1	2	3	4		1	2	3	4
1		105.8	110.8	111.8	1		110.5	111.7	106.1
2			110.8	109.7	2			108.6	110.9
j 3				108.0	j 3				109.0
ϕ			120.1	238.8	ϕ			120.9	239.8

bond lengths P – Ci

P1				P2			
1.789	1.784	1.792	1.808	1.789	1.793	1.797	1.781

Secondary bonds	d	dv	Δd	angles	Ci —— P ---- X		
P1 ---- Cl	6.61	3.55	3.06	106.1	66.1	142.0	47.6
P1 ---- Cl	7.47	3.55	3.92	67.8	173.4	74.0	72.3
P2 ---- Cl	7.75	3.55	4.20	127.4	107.8	19.4	92.0
P2 ---- Cl	6.16	3.55	2.61	95.1	61.8	59.6	158.7

35. TPHCLC[154]: TETRAPHENYLPHOSPHONIUM TRICHLOROCUPRATE(II)



a = 9.226 Å
b = 19.301Å
c = 13.842Å
β = 111.3°
$P2_1/c$
Z = 4

angles Cj — P — Ci

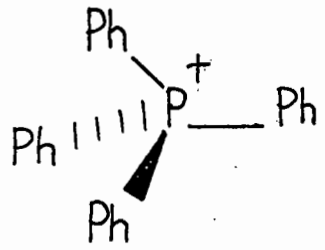
	i 1	2	3	4
1		111.9	110.0	107.3
2			108.0	108.8
j 3				110.8
ϕ			120.0	240.8

bond lengths P — Ci

1.777	1.783	1.769	1.768

Secondary bonds	d	dv	Δd	angles	Ci —— P ---- X		
P ---- Cl	7.64	3.55	4.09	137.3	25.6	92.0	97.4
P ---- Cl	5.33	3.55	1.78	61.8	171.7	80.0	69.7
P ---- Cl	5.59	3.55	2.04	54.7	103.5	61.7	147.4

36. PPHTCM[155]: TETRAPHENYLPHOSPHONIUM TRITHIOCYANATOMERCURATE(II)



$Hg(SCN)_3^-$

a = 11.574 Å
b = 20.014 Å
c = 12.144 Å
β = 103.56°
$P2_1/c$
Z = 4

angles Cj – P – Ci

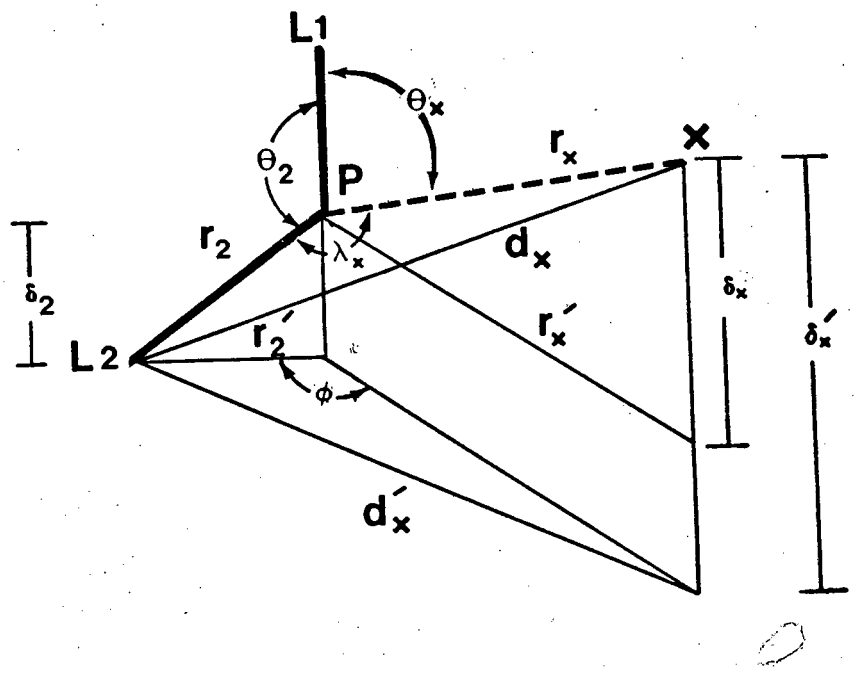
	i 1	2	3	4
1		110.8	108.9	106.9
2			108.1	110.3
j 3				111.9
ϕ			118.8	239.8

bond lengths P – Ci

1.792	1.794	1.790	1.785

Secondary bonds	d	dv	Δd	angles	Ci — P ---- X		
P ---- S1	4.917	3.60	1.32	48.6	158.5	78.2	85.0
P ---- N3	4.518	3.35	1.17	162.3	76.3	53.6	84.7

APPENDIX THREE



C1 is fixed at the north pole, $\theta = 0$

C2 is fixed at $\phi = 0$

for any atom X,

$$r_x' = r_x \sin\theta_x \qquad r_2' = r_2 \sin\theta_2$$

$$d_x^2 = r_2^2 + r_x^2 - 2r_2 r_x \cos\lambda_x$$

$$\delta_x = r_x \cos\theta_x \qquad \delta_2 = -r_2 \cos\theta_2$$

$$\delta_x' = \delta_x + \delta_2$$

$$(d_x')^2 = d_x^2 - (\delta_x')^2$$

$$\cos = \frac{(r_2')^2 + (r_x')^2 - (d_x')^2}{2r_2' r_x'}$$

An Apple II computer was used for computations.

APPENDIX FOUR

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	2	1	36	35	-3	3	1	77	76	-1	4	1	24	-23	6	5	1	8	8	-15	7	1	7	-4
-2	2	1	38	38	-2	3	1	14	-14	0	4	1	12	-11	7	5	1	22	21	-14	7	1	15	-16
-1	2	1	271	309	-1	3	1	40	39	1	4	1	59	59	8	5	1	11	10	-12	7	1	9	-10
0	2	1	54	-54	0	3	1	25	27	2	4	1	30	-29	9	5	1	17	-17	-10	7	1	9	9
1	2	1	39	-40	1	3	1	118	-121	3	4	1	9	-11	11	5	1	10	-11	-9	7	1	26	26
2	2	1	39	38	2	3	1	44	-44	4	4	1	20	19	12	5	1	14	-12	-8	7	1	6	-6
3	2	1	83	-86	3	3	1	24	25	5	4	1	10	11	13	5	1	26	25	-7	7	1	15	-14
4	2	1	50	49	4	3	1	7	6	6	4	1	5	-4	15	5	1	9	-10	-5	7	1	29	-30
5	2	1	19	-20	5	3	1	52	-50	7	4	1	52	52	17	5	1	21	-20	-4	7	1	7	7
6	2	1	25	-22	6	3	1	6	10	8	4	1	6	-4	-15	6	1	9	-7	-3	7	1	45	44
7	2	1	33	33	7	3	1	18	18	9	4	1	16	-16	-14	6	1	8	-7	-2	7	1	9	10
9	2	1	6	7	8	3	1	48	47	14	4	1	7	-5	-11	6	1	16	-16	-1	7	1	22	-22
10	2	1	19	17	9	3	1	45	43	15	4	1	10	11	-10	6	1	26	26	1	7	1	40	-40
11	2	1	14	-16	10	3	1	17	-16	16	4	1	15	-16	-8	6	1	19	-19	2	7	1	13	-15
13	2	1	42	-39	12	3	1	32	-32	17	4	1	14	-15	-7	6	1	7	6	3	7	1	21	22
14	2	1	14	13	13	3	1	9	-8	18	4	1	8	-7	-6	6	1	38	-39	4	7	1	32	-33
15	2	1	25	27	15	3	1	18	19	-17	5	1	8	11	-5	6	1	35	-34	5	7	1	18	17
16	2	1	11	13	16	3	1	25	25	-15	5	1	22	-22	-4	6	1	17	17	7	7	1	19	19
17	2	1	10	21	17	3	1	15	-15	-11	5	1	17	17	-3	6	1	20	19	8	7	1	37	37
-18	3	1	7	2	18	3	1	18	18	-10	5	1	7	-8	-2	6	1	10	13	9	7	1	26	-25
-17	3	1	12	15	-18	4	1	14	14	-9	5	1	24	-23	-1	6	1	30	31	10	7	1	13	-13
-16	3	1	13	14	-17	4	1	23	22	-8	5	1	43	43	1	6	1	39	-38	11	7	1	13	-15
-15	3	1	9	9	-16	4	1	8	5	-7	5	1	23	-24	2	6	1	30	-29	12	7	1	22	-22
-14	3	1	23	-23	-15	4	1	13	12	-6	5	1	12	-13	3	6	1	59	-60	13	7	1	9	9
-13	3	1	12	10	-13	4	1	21	-22	-5	5	1	8	9	4	6	1	21	20	-13	8	1	12	-10
-12	3	1	6	3	-12	4	1	28	28	-4	5	1	19	-19	5	6	1	30	-30	-12	8	1	13	13
-11	3	1	29	-28	-10	4	1	10	-13	-2	5	1	49	51	6	6	1	28	27	-10	8	1	18	16
-10	3	1	12	13	-9	4	1	62	60	-1	5	1	49	49	7	6	1	29	29	-9	8	1	23	24
-9	3	1	12	-11	-8	4	1	44	-41	0	5	1	26	-25	8	6	1	23	-21	-8	8	1	11	-9
-8	3	1	11	10	-7	4	1	28	-26	1	5	1	42	-43	9	6	1	6	8	-6	8	1	8	10
-7	3	1	34	32	-6	4	1	19	-18	2	5	1	22	22	10	6	1	20	-21	-4	8	1	11	8
-6	3	1	28	-28	-4	4	1	25	-25	3	5	1	36	-37	11	6	1	7	-8	-3	8	1	10	8
-5	3	1	6	5	-3	4	1	15	-15	4	5	1	21	21	13	6	1	19	-17	-1	8	1	17	-19
-4	3	1	38	-37	-2	4	1	24	26	5	5	1	20	19	14	6	1	25	26	0	8	1	22	22

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	8	1	26	27	5	10	1	9	-8	-3	1	2	123	-121	-1	2	2	52	50	8	3	2	8	8
2	8	1	22	-22	-20	0	2	14	-14	-2	1	2	27	-26	0	2	2	14	-14	11	3	2	29	-29
3	8	1	21	-20	-16	0	2	11	12	-1	1	2	88	91	3	2	2	12	12	12	3	2	18	20
5	8	1	7	-5	-14	0	2	15	15	0	1	2	49	-53	4	2	2	38	-40	13	3	2	16	-17
6	8	1	37	38	-12	0	2	16	-15	1	1	2	6	2	5	2	2	11	7	14	3	2	13	-10
7	8	1	32	33	-10	0	2	46	-46	2	1	2	80	85	6	2	2	36	37	15	3	2	23	20
8	8	1	19	-20	-8	0	2	23	24	3	1	2	78	-81	7	2	2	36	-36	16	3	2	24	-25
9	8	1	8	10	-6	0	2	61	-56	4	1	2	13	-12	8	2	2	28	28	-17	4	2	9	10
10	8	1	27	-27	-4	0	2	54	49	5	1	2	54	55	9	2	2	6	-8	-14	4	2	8	9
11	8	1	15	-14	-2	0	2	248	269	6	1	2	50	-52	11	2	2	6	-5	-12	4	2	13	-12
12	8	1	18	18	0	0	2	14	-12	7	1	2	61	63	12	2	2	10	8	-11	4	2	23	22
-8	9	1	9	8	2	0	2	77	-77	8	1	2	48	49	13	2	2	18	-18	-10	4	2	19	-20
-7	9	1	13	-13	4	0	2	48	47	9	1	2	41	-47	14	2	2	16	17	-9	4	2	26	-25
-6	9	1	18	17	6	0	2	27	-28	10	1	2	17	-22	16	2	2	29	-28	-8	4	2	19	19
-5	9	1	15	15	8	0	2	55	-54	11	1	2	48	-48	19	2	2	10	-10	-6	4	2	21	-20
-4	9	1	34	-36	10	0	2	6	-5	14	1	2	35	36	-18	3	2	8	6	-5	4	2	65	66
-3	9	1	10	12	12	0	2	69	-68	15	1	2	45	44	-17	3	2	7	-7	-4	4	2	74	73
1	9	1	16	-15	14	0	2	44	45	16	1	2	26	-27	-16	3	2	13	-11	-3	4	2	27	26
2	9	1	19	-19	16	0	2	39	39	19	1	2	13	-14	-12	3	2	15	16	-2	4	2	73	75
4	9	1	8	6	18	0	2	29	-27	-19	2	2	9	-9	-10	3	2	64	-61	-1	4	2	32	30
5	9	1	23	22	-19	1	2	27	-28	-18	2	2	18	17	-9	3	2	57	56	0	4	2	88	-88
6	9	1	8	6	-17	1	2	16	17	-16	2	2	14	14	-6	3	2	22	22	1	4	2	8	9
9	9	1	12	-11	-16	1	2	7	-6	-14	2	2	21	-19	-5	3	2	6	4	2	4	2	50	-49
10	9	1	22	22	-15	1	2	9	8	-13	2	2	26	27	-4	3	2	10	-11	3	4	2	13	-13
-6	10	1	21	-19	-14	1	2	20	20	-12	2	2	8	-8	-3	3	2	12	-12	4	4	2	38	-38
-5	10	1	18	-17	-13	1	2	26	-25	-10	2	2	58	53	-2	3	2	100	101	6	4	2	23	21
-4	10	1	10	10	-12	1	2	18	-18	-9	2	2	51	-49	-1	3	2	24	25	8	4	2	27	27
-3	10	1	18	16	-10	1	2	36	35	-8	2	2	42	-41	0	3	2	79	-80	11	4	2	13	14
-2	10	1	14	10	-9	1	2	38	39	-7	2	2	7	-8	1	3	2	33	31	12	4	2	22	-22
-1	10	1	12	12	-8	1	2	36	34	-6	2	2	6	-4	2	3	2	26	-28	13	4	2	25	24
0	10	1	22	-20	-7	1	2	28	-29	-5	2	2	64	-61	3	3	2	5	-5	14	4	2	22	20
1	10	1	10	13	-6	1	2	67	-65	-4	2	2	99	-97	4	3	2	11	-9	15	4	2	10	10
2	10	1	9	-8	-5	1	2	25	-26	-3	2	2	161	-159	5	3	2	6	-5	16	4	2	19	19
4	10	1	14	14	-4	1	2	21	-18	-2	2	2	21	21	7	3	2	40	40	-17	5	2	14	12

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-15	5	2	15	-13	-3	6	2	28	27	8	7	2	32	-34	4	9	2	11	9	-4	1	3	60	-60
-13	5	2	12	14	-1	6	2	33	35	9	7	2	18	18	6	9	2	19	19	-3	1	3	24	21
-12	5	2	15	-16	0	6	2	27	27	10	7	2	8	-7	7	9	2	17	16	-2	1	3	43	42
-10	5	2	42	40	1	6	2	44	-45	11	7	2	20	-21	8	9	2	18	-17	-1	1	3	29	-28
-9	5	2	33	-32	2	6	2	11	-11	13	7	2	15	-14	9	9	2	9	-9	0	1	3	38	39
-8	5	2	18	-19	3	6	2	21	21	-12	8	2	23	-22	-4	10	2	9	-6	1	1	3	48	45
-7	5	2	11	-10	4	6	2	10	-9	-10	8	2	10	-9	-1	10	2	10	9	2	1	3	21	17
-6	5	2	21	-23	5	6	2	13	14	-9	8	2	13	13	0	10	2	15	14	3	1	3	9	-9
-5	5	2	14	13	6	6	2	37	39	-8	8	2	28	26	-17	0	3	16	14	4	1	3	18	-20
-4	5	2	50	51	7	6	2	15	-15	-7	8	2	33	-33	-11	0	3	37	38	5	1	3	6	4
-2	5	2	7	6	8	6	2	19	19	-6	8	2	24	-23	-9	0	3	18	-17	6	1	3	51	49
-1	5	2	14	14	9	6	2	22	-22	-4	8	2	15	14	-7	0	3	52	49	7	1	3	64	-63
0	5	2	31	-31	10	6	2	23	-21	-3	8	2	22	23	-5	0	3	107	-100	8	1	3	19	-16
1	5	2	9	-10	13	6	2	9	12	-2	8	2	17	17	-3	0	3	73	-72	9	1	3	19	20
2	5	2	15	15	14	6	2	12	-12	-1	8	2	33	-34	-1	0	3	138	138	10	1	3	48	-50
3	5	2	39	-39	-14	7	2	19	-18	0	8	2	28	-28	1	0	3	43	40	11	1	3	21	20
6	5	2	16	16	-13	7	2	9	-9	1	8	2	20	-18	3	0	3	78	-77	12	1	3	9	-7
7	5	2	16	17	-12	7	2	17	17	2	8	2	15	-14	5	0	3	37	36	13	1	3	27	-25
8	5	2	6	-6	-11	7	2	18	-20	3	8	2	17	16	7	0	3	58	57	14	1	3	48	49
9	5	2	9	-8	-9	7	2	16	18	4	8	2	11	-13	9	0	3	9	7	15	1	3	21	21
11	5	2	11	-9	-8	7	2	9	-7	6	8	2	16	16	11	0	3	19	21	18	1	3	19	-18
14	5	2	29	29	-7	7	2	14	15	7	8	2	11	10	13	0	3	7	8	-17	2	3	16	17
15	5	2	19	18	-6	7	2	16	17	8	8	2	10	11	15	0	3	11	11	-13	2	3	41	-41
16	5	2	22	-21	-5	7	2	30	-31	9	8	2	19	-20	17	0	3	10	9	-12	2	3	28	28
-16	6	2	15	16	-4	7	2	8	-8	10	8	2	12	-12	-17	1	3	15	-15	-11	2	3	20	-19
-15	6	2	18	-18	-2	7	2	23	23	11	8	2	9	-9	-16	1	3	13	13	-9	2	3	33	32
-14	6	2	16	-16	-1	7	2	6	-7	-10	9	2	15	14	-12	1	3	30	-29	-8	2	3	8	-7
-13	6	2	16	16	0	7	2	20	-20	-7	9	2	13	14	-11	1	3	34	-34	-7	2	3	26	24
-12	6	2	8	-9	1	7	2	7	-7	-5	9	2	9	9	-10	1	3	72	68	-6	2	3	33	32
-10	6	2	16	16	2	7	2	21	-21	-2	9	2	31	-30	-9	1	3	22	-19	-5	2	3	52	54
-8	6	2	14	-14	3	7	2	14	13	-1	9	2	8	11	-8	1	3	39	40	-2	2	3	51	48
-7	6	2	15	-15	4	7	2	33	33	0	9	2	10	-12	-7	1	3	35	36	-1	2	3	26	-27
-6	6	2	6	8	6	7	2	27	27	1	9	2	16	16	-6	1	3	29	-34	0	2	3	46	45
-5	6	2	18	-19	7	7	2	10	9	3	9	2	27	-27	-5	1	3	12	-12	1	2	3	41	-42

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	2	3	61	57	7	3	3	12	14	17	4	3	12	13	-10	6	3	25	25	12	7	3	12	13
4	2	3	51	-51	8	3	3	26	-26	-17	5	3	11	-10	-9	6	3	33	35	13	7	3	22	21
5	2	3	26	27	9	3	3	18	18	-16	5	3	21	19	-8	6	3	17	-18	-11	8	3	9	8
6	2	3	35	-36	10	3	3	14	-13	-15	5	3	9	-10	-7	6	3	8	-7	-10	8	3	14	15
7	2	3	32	-32	11	3	3	8	9	-13	5	3	8	9	-6	6	3	14	15	-9	8	3	11	-9
8	2	3	14	13	13	3	3	19	20	-12	5	3	17	-17	-5	6	3	28	27	-8	8	3	9	-9
11	2	3	59	-58	14	3	3	21	21	-11	5	3	12	-12	-3	6	3	31	30	-7	8	3	15	15
12	2	3	13	14	15	3	3	27	-26	-10	5	3	29	28	-1	6	3	53	-51	-5	8	3	9	-9
13	2	3	29	29	16	3	3	15	14	-9	5	3	23	23	0	6	3	14	-13	-4	8	3	9	9
14	2	3	21	25	17	3	3	8	7	-8	5	3	14	-14	1	6	3	29	-29	-3	8	3	9	-6
15	2	3	30	31	-18	4	3	14	14	-7	5	3	18	16	2	6	3	7	-9	-2	8	3	32	33
17	2	3	21	-20	-17	4	3	16	15	-6	5	3	20	-21	4	6	3	12	10	-1	8	3	30	30
-18	3	3	16	15	-16	4	3	13	-10	-5	5	3	20	-22	6	6	3	28	29	0	8	3	15	-16
-16	3	3	8	8	-15	4	3	19	-18	-4	5	3	26	26	7	6	3	13	11	1	8	3	7	-5
-14	3	3	14	-13	-14	4	3	17	16	-3	5	3	49	50	8	6	3	39	-39	2	8	3	10	-11
-13	3	3	12	13	-13	4	3	6	4	-2	5	3	8	8	9	6	3	12	13	3	8	3	13	-15
-12	3	3	11	-9	-10	4	3	16	-15	-1	5	3	45	-45	12	6	3	22	22	4	8	3	24	24
-11	3	3	40	40	-9	4	3	37	-37	0	5	3	16	16	13	6	3	21	22	5	8	3	19	17
-10	3	3	9	-7	-8	4	3	24	-24	1	5	3	28	-28	-13	7	3	18	-18	8	8	3	20	-20
-9	3	3	48	-47	-6	4	3	39	-40	2	5	3	31	31	-11	7	3	33	33	10	8	3	16	15
-8	3	3	17	-17	-5	4	3	37	-39	3	5	3	14	14	-10	7	3	13	-16	-9	9	3	13	11
-7	3	3	30	-31	-4	4	3	34	-33	4	5	3	41	-43	-8	7	3	38	40	-8	9	3	16	15
-6	3	3	12	-13	-3	4	3	45	46	5	5	3	21	22	-7	7	3	30	-29	-6	9	3	34	-35
-5	3	3	29	28	-2	4	3	72	72	6	5	3	16	16	-6	7	3	8	8	-4	9	3	10	-9
-4	3	3	39	-40	0	4	3	65	-65	7	5	3	29	-28	-5	7	3	8	-11	-3	9	3	21	19
-3	3	3	14	-15	1	4	3	43	-44	8	5	3	12	14	-3	7	3	16	-16	1	9	3	9	-3
-2	3	3	8	-4	5	4	3	17	15	9	5	3	7	-6	-1	7	3	12	-13	2	9	3	13	13
-1	3	3	35	34	6	4	3	29	-30	10	5	3	23	-23	0	7	3	19	18	3	9	3	8	7
1	3	3	40	40	7	4	3	20	19	13	5	3	7	6	2	7	3	26	-25	4	9	3	13	-15
2	3	3	45	-45	8	4	3	13	13	-15	6	3	10	11	4	7	3	7	-4	5	9	3	13	14
3	3	3	14	-15	13	4	3	36	-37	-14	6	3	24	-27	5	7	3	30	29	7	9	3	9	-6
4	3	3	22	21	14	4	3	13	13	-13	6	3	23	-23	6	7	3	23	23	-2	10	3	11	-13
5	3	3	48	-48	15	4	3	26	-28	-12	6	3	10	10	9	7	3	26	-27	-18	0	4	14	13
6	3	3	25	25	16	4	3	23	-23	-11	6	3	10	-12	10	7	3	18	-16	-16	0	4	15	15

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-14	0	4	49	-50	6	1	4	33	30	15	2	4	8	-11	-10	4	4	38	38	-1	5	4	10	10
-12	0	4	10	-9	7	1	4	44	-42	16	2	4	21	22	-9	4	4	21	-20	0	5	4	22	21
-10	0	4	38	41	10	1	4	7	-5	-18	3	4	18	-15	-8	4	4	8	8	1	5	4	33	-33
-8	0	4	23	21	11	1	4	13	-14	-17	3	4	14	14	-7	4	4	30	29	2	5	4	9	-9
-6	0	4	37	-36	12	1	4	20	20	-16	3	4	8	7	-6	4	4	50	51	3	5	4	13	13
-4	0	4	65	-64	13	1	4	15	14	-15	3	4	9	-7	-5	4	4	11	-11	5	5	4	12	-13
-2	0	4	9	-10	14	1	4	22	-21	-13	3	4	25	-26	-3	4	4	40	41	6	5	4	19	20
0	0	4	54	53	15	1	4	22	23	-12	3	4	10	-9	-2	4	4	55	-55	8	5	4	30	-31
2	0	4	24	22	16	1	4	9	11	-11	3	4	26	26	-1	4	4	6	4	10	5	4	13	-11
4	0	4	53	-53	17	1	4	18	-16	-10	3	4	22	21	2	4	4	18	-19	11	5	4	11	11
6	0	4	48	-45	-18	2	4	12	12	-9	3	4	32	32	3	4	4	16	-16	12	5	4	18	19
8	0	4	15	16	-17	2	4	10	-10	-5	3	4	8	-9	4	4	4	11	-10	13	5	4	26	26
10	0	4	71	-70	-16	2	4	7	-7	-4	3	4	18	18	5	4	4	13	14	14	5	4	15	-15
12	0	4	23	24	-13	2	4	20	21	-3	3	4	44	45	6	4	4	21	21	-15	6	4	7	-6
14	0	4	30	28	-12	2	4	28	-29	-2	3	4	20	-20	7	4	4	35	-33	-11	6	4	14	16
16	0	4	28	-28	-10	2	4	23	22	-1	3	4	13	13	8	4	4	10	11	-10	6	4	24	-25
-18	1	4	9	10	-9	2	4	16	-14	1	3	4	9	11	9	4	4	15	12	-9	6	4	10	-11
-17	1	4	20	20	-8	2	4	31	32	3	3	4	15	-15	11	4	4	12	13	-7	6	4	26	-25
-15	1	4	16	-16	-7	2	4	28	-27	4	3	4	22	-23	12	4	4	31	32	-5	6	4	11	-11
-14	1	4	22	25	-6	2	4	37	-36	5	3	4	6	6	13	4	4	12	11	-3	6	4	46	45
-13	1	4	24	-24	-5	2	4	43	-43	6	3	4	43	-44	15	4	4	18	-19	-2	6	4	14	13
-12	1	4	44	45	-4	2	4	12	-12	7	3	4	15	14	-16	5	4	15	-15	-1	6	4	26	-25
-11	1	4	34	34	-3	2	4	48	48	8	3	4	29	29	-14	5	4	10	-9	0	6	4	8	8
-10	1	4	24	-25	-2	2	4	17	20	9	3	4	29	-28	-12	5	4	28	28	1	6	4	7	-9
-8	1	4	13	-13	-1	2	4	35	-36	10	3	4	13	11	-11	5	4	26	-26	2	6	4	31	-31
-6	1	4	9	9	2	2	4	10	-11	11	3	4	8	-11	-10	5	4	18	-19	3	6	4	16	17
-5	1	4	79	-79	3	2	4	46	42	13	3	4	21	20	-9	5	4	26	27	4	6	4	9	9
-3	1	4	40	-40	4	2	4	26	26	14	3	4	13	13	-8	5	4	36	-36	6	6	4	8	9
-2	1	4	44	41	5	2	4	16	-15	16	3	4	13	-15	-7	5	4	9	-9	7	6	4	9	-9
-1	1	4	76	74	6	2	4	57	56	-16	4	4	13	13	-6	5	4	16	-16	8	6	4	9	-9
0	1	4	13	12	9	2	4	22	21	-15	4	4	15	-15	-5	5	4	43	-43	11	6	4	11	-14
1	1	4	11	-12	10	2	4	7	-7	-14	4	4	41	-40	-4	5	4	16	-16	12	6	4	37	-37
4	1	4	22	-21	12	2	4	33	-34	-13	4	4	13	13	-3	5	4	45	46	13	6	4	25	24
5	1	4	29	28	14	2	4	8	10	-11	4	4	25	25	-2	5	4	24	23	-13	7	4	13	-13

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-10	7	4	37	38	1	9	4	11	-10	3	1	5	20	-19	-14	3	5	19	-20	2	4	5	19	21
-8	7	4	8	10	2	9	4	10	-9	5	1	5	44	-43	-13	3	5	26	26	3	4	5	22	21
-7	7	4	36	-38	4	9	4	14	12	6	1	5	12	10	-12	3	5	13	13	5	4	5	9	-11
-6	7	4	16	-15	-15	0	5	11	11	7	1	5	21	21	-11	3	5	41	-44	7	4	5	20	22
-5	7	4	14	-15	-13	0	5	49	-50	10	1	5	20	-19	-10	3	5	15	15	8	4	5	17	17
-4	7	4	21	21	-11	0	5	40	39	11	1	5	8	-4	-9	3	5	12	-12	9	4	5	9	-9
-3	7	4	12	11	-9	0	5	45	47	13	1	5	18	19	-6	3	5	43	-42	10	4	5	16	-16
0	7	4	22	-20	-7	0	5	43	-43	14	1	5	19	19	-5	3	5	13	-11	11	4	5	49	-49
1	7	4	17	19	-5	0	5	19	-20	15	1	5	16	-17	-4	3	5	15	-14	12	4	5	24	24
2	7	4	13	11	-3	0	5	32	32	16	1	5	10	-11	-3	3	5	48	48	-14	5	5	10	-6
3	7	4	25	-26	-1	0	5	42	42	-17	2	5	13	12	-2	3	5	28	29	-13	5	5	9	9
4	7	4	25	26	1	0	5	27	-26	-16	2	5	8	-6	-1	3	5	17	17	-12	5	5	16	15
6	7	4	15	-12	3	0	5	12	12	-15	2	5	44	-45	0	3	5	11	-11	-11	5	5	57	60
7	7	4	30	31	5	0	5	42	40	-12	2	5	22	23	1	3	5	20	-21	-10	5	5	8	9
8	7	4	12	-10	9	0	5	11	-10	-11	2	5	29	29	2	3	5	9	-8	-8	5	5	41	-42
9	7	4	27	-27	11	0	5	29	-30	-10	2	5	19	-20	3	3	5	16	16	-7	5	5	15	-15
10	7	4	9	-12	13	0	5	23	24	-9	2	5	28	28	4	3	5	14	13	-5	5	5	8	6
11	7	4	14	-12	15	0	5	33	34	-8	2	5	8	9	5	3	5	10	10	-4	5	5	14	15
-10	8	4	9	9	-18	1	5	17	16	-6	2	5	7	7	7	3	5	24	-26	-3	5	5	22	22
-9	8	4	15	-18	-15	1	5	12	-14	-5	2	5	51	-50	8	3	5	14	15	-2	5	5	21	20
-5	8	4	8	9	-14	1	5	32	-34	-4	2	5	15	-16	10	3	5	10	10	-1	5	5	13	-12
-2	8	4	13	-12	-13	1	5	19	-19	-3	2	5	25	-26	14	3	5	14	10	0	5	5	24	24
0	8	4	8	7	-12	1	5	11	11	-1	2	5	27	27	-13	4	5	18	-19	1	5	5	7	7
1	8	4	9	-10	-11	1	5	28	27	0	2	5	26	26	-12	4	5	12	13	2	5	5	31	-32
3	8	4	23	-23	-10	1	5	33	34	2	2	5	15	-17	-10	4	5	18	-20	4	5	5	8	-8
4	8	4	20	-19	-9	1	5	17	-16	3	2	5	43	-45	-9	4	5	24	23	5	5	5	16	-16
5	8	4	30	32	-7	1	5	6	-4	5	2	5	19	20	-8	4	5	19	-19	6	5	5	13	13
7	8	4	11	-6	-6	1	5	28	-29	6	2	5	27	-26	-7	4	5	27	-26	7	5	5	8	6
8	8	4	18	17	-5	1	5	35	-34	7	2	5	16	15	-6	4	5	15	-16	8	5	5	34	-33
-7	9	4	9	6	-4	1	5	16	-15	8	2	5	9	-9	-5	4	5	13	-12	11	5	5	15	17
-5	9	4	7	-7	-3	1	5	11	-11	9	2	5	25	-24	-4	4	5	37	37	12	5	5	9	5
-2	9	4	7	-3	-2	1	5	71	70	11	2	5	8	7	-3	4	5	17	16	13	5	5	23	24
-1	9	4	15	16	1	1	5	23	21	-17	3	5	15	-13	-2	4	5	28	-26	-13	6	5	8	6
0	9	4	8	-10	2	1	5	48	-46	-15	3	5	12	13	-1	4	5	11	-9	-12	6	5	29	31

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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-10	6	5	10	-3	-2	8	5	14	-15	2	1	6	36	37	14	2	6	27	26	2	4	6	46	-47
-9	6	5	13	13	-1	8	5	10	10	4	1	6	13	13	-15	3	6	17	-17	3	4	6	17	18
-8	6	5	13	14	0	8	5	14	-16	5	1	6	30	32	-12	3	6	54	55	4	4	6	24	22
-7	6	5	8	-5	1	8	5	16	-15	6	1	6	50	-50	-11	3	6	20	22	5	4	6	16	-17
-6	6	5	17	-18	-1	9	5	15	-13	7	1	6	27	28	-10	3	6	25	-25	6	4	6	15	14
-4	6	5	23	22	-16	0	6	20	-19	8	1	6	11	7	-9	3	6	21	-22	9	4	6	14	-16
-2	6	5	11	10	-12	0	6	19	21	9	1	6	10	-7	-8	3	6	14	-13	10	4	6	17	15
-1	6	5	8	7	-10	0	6	11	9	11	1	6	19	-17	-7	3	6	18	-18	12	4	6	10	-10
0	6	5	24	-24	-8	0	6	7	8	13	1	6	17	17	-6	3	6	10	8	-13	5	6	7	9
1	6	5	8	9	-6	0	6	46	-44	14	1	6	16	15	-5	3	6	12	13	-11	5	6	18	18
2	6	5	14	-14	-4	0	6	40	-40	-14	2	6	35	-33	-3	3	6	30	30	-10	5	6	32	-35
3	6	5	40	-41	-2	0	6	63	61	-13	2	6	16	20	-2	3	6	20	-18	-9	5	6	11	-14
4	6	5	32	32	0	0	6	11	10	-12	2	6	17	18	0	3	6	19	21	-8	5	6	12	-12
5	6	5	9	5	2	0	6	68	-67	-11	2	6	9	-10	1	3	6	27	-29	-7	5	6	36	-36
6	6	5	12	-11	4	0	6	43	42	-10	2	6	42	42	2	3	6	24	-24	-5	5	6	28	29
7	6	5	40	41	6	0	6	21	22	-9	2	6	14	14	3	3	6	22	-23	-4	5	6	23	21
8	6	5	10	7	8	0	6	12	-10	-8	2	6	26	-26	4	3	6	9	-6	-3	5	6	25	24
11	6	5	18	18	10	0	6	17	-17	-6	2	6	9	-11	6	3	6	15	15	-2	5	6	15	15
-11	7	5	9	6	12	0	6	15	-20	-5	2	6	9	-5	7	3	6	14	-14	0	5	6	29	-29
-10	7	5	16	17	14	0	6	17	16	-3	2	6	17	-19	10	3	6	22	23	2	5	6	15	17
-9	7	5	38	-39	-16	1	6	10	7	-2	2	6	19	19	12	3	6	10	12	3	5	6	22	-24
-7	7	5	19	-19	-15	1	6	19	-18	0	2	6	8	-9	-14	4	6	8	8	4	5	6	33	33
-6	7	5	19	-21	-14	1	6	14	15	1	2	6	44	44	-13	4	6	19	20	6	5	6	47	-46
-4	7	5	25	25	-12	1	6	16	-17	2	2	6	12	13	-12	4	6	11	13	7	5	6	17	15
-2	7	5	18	20	-11	1	6	22	21	3	2	6	23	23	-10	4	6	8	8	8	5	6	10	-9
1	7	5	20	-20	-10	1	6	11	14	5	2	6	22	-23	-9	4	6	8	7	9	5	6	16	18
2	7	5	7	-6	-9	1	6	16	16	6	2	6	28	28	-8	4	6	9	-10	10	5	6	8	-7
3	7	5	11	13	-8	1	6	17	19	7	2	6	26	27	-7	4	6	8	-9	11	5	6	9	9
5	7	5	39	39	-7	1	6	18	-21	8	2	6	15	-14	-6	4	6	29	-31	-10	6	6	20	19
7	7	5	33	-32	-6	1	6	15	14	9	2	6	12	-13	-3	4	6	12	13	-9	6	6	16	-16
-8	8	5	17	-17	-5	1	6	27	-25	10	2	6	41	-43	-2	4	6	34	34	-7	6	6	12	-14
-5	8	5	8	-5	-4	1	6	7	7	11	2	6	9	4	-1	4	6	25	-24	-6	6	6	24	-26
-4	8	5	37	39	-3	1	6	43	42	12	2	6	15	14	0	4	6	15	-14	-5	6	6	34	34

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	6	6	24	24	-14	1	7	16	-16	-12	3	7	16	16	-10	5	7	7	-6	-10	1	8	18	20
-3	6	6	15	-13	-13	1	7	29	28	-11	3	7	12	-16	-8	5	7	10	-13	-6	1	8	9	9
-2	6	6	17	17	-12	1	7	12	11	-9	3	7	12	13	-7	5	7	10	12	-4	1	8	7	6
0	6	6	17	-19	-11	1	7	17	-17	-8	3	7	8	-6	-6	5	7	15	-13	-3	1	8	13	11
1	6	6	10	-12	-9	1	7	25	-26	-7	3	7	11	-9	-5	5	7	14	15	-2	1	8	15	-15
2	6	6	8	8	-8	1	7	10	-9	-5	3	7	18	14	-4	5	7	23	24	-1	1	8	15	-17
3	6	6	9	11	-5	1	7	20	-20	-4	3	7	32	34	-3	5	7	14	-16	0	1	8	20	19
4	6	6	16	-14	-4	1	7	16	16	-3	3	7	12	9	-2	5	7	15	13	1	1	8	19	-18
5	6	6	12	13	-1	1	7	18	18	-1	3	7	19	-17	0	5	7	20	-19	2	1	8	21	-23
6	6	6	11	11	0	1	7	26	-26	0	3	7	18	-19	1	5	7	21	-22	3	1	8	22	22
7	6	6	9	-9	1	1	7	15	-15	1	3	7	36	37	3	5	7	12	13	4	1	8	45	-43
9	6	6	26	-25	2	1	7	20	-22	2	3	7	8	-10	4	5	7	8	-9	5	1	8	24	24
-9	7	6	8	-9	3	1	7	14	14	3	3	7	9	9	5	5	7	13	11	6	1	8	11	9
-8	7	6	8	-6	4	1	7	19	19	4	3	7	18	18	-7	6	7	12	-14	7	1	8	12	15
-4	7	6	25	26	5	1	7	22	21	5	3	7	63	-64	-6	6	7	11	11	9	1	8	20	-19
-2	7	6	13	-14	10	1	7	20	-21	6	3	7	31	31	-4	6	7	9	9	-10	2	8	17	-15
-1	7	6	13	15	-14	2	7	13	10	7	3	7	9	10	-3	6	7	13	13	-8	2	8	11	-11
0	7	6	10	-11	-12	2	7	8	10	8	3	7	17	19	-2	6	7	26	-24	-6	2	8	9	13
1	7	6	9	-10	-11	2	7	11	9	9	3	7	8	-9	0	6	7	20	-19	-3	2	8	12	-11
5	7	6	18	17	-8	2	7	8	-8	10	3	7	11	-11	1	6	7	24	-23	0	2	8	10	-10
-2	8	6	13	12	-7	2	7	20	-18	-11	4	7	34	32	2	6	7	20	20	1	2	8	13	13
-1	8	6	19	-19	-6	2	7	11	-13	-10	4	7	8	-2	4	6	7	10	10	2	2	8	25	-24
1	8	6	19	-20	-5	2	7	22	-20	-9	4	7	24	-25	5	6	7	16	16	4	2	8	37	38
2	8	6	18	-18	-3	2	7	49	49	-7	4	7	18	-17	-2	7	7	9	2	6	2	8	13	-13
-11	0	7	40	40	-1	2	7	12	-12	-5	4	7	26	26	-1	7	7	10	-10	8	2	8	23	-20
-9	0	7	21	-21	0	2	7	9	7	-3	4	7	9	8	0	7	7	8	5	-10	3	8	11	-11
-7	0	7	13	-13	1	2	7	49	-48	2	4	7	16	19	1	7	7	10	7	-9	3	8	11	-10
-3	0	7	16	16	2	2	7	15	13	3	4	7	19	-19	-12	0	8	14	15	-8	3	8	22	23
-1	0	7	8	6	3	2	7	36	37	4	4	7	12	12	-6	0	8	28	-29	-6	3	8	11	-11
3	0	7	23	-23	5	2	7	23	23	5	4	7	34	35	-4	0	8	49	50	-5	3	8	13	15
5	0	7	31	29	6	2	7	15	-15	7	4	7	12	-14	0	0	8	24	-25	-4	3	8	8	-8
7	0	7	14	-15	7	2	7	8	-6	8	4	7	18	-19	2	0	8	15	14	-3	3	8	8	-3
9	0	7	37	-36	8	2	7	22	-22	9	4	7	22	-18	4	0	8	29	28	-1	3	8	12	-13
11	0	7	8	6	11	2	7	22	-23	10	4	7	14	14	6	0	8	8	-6	0	3	8	24	-24

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	0	123	-124	14	2	0	18	18	18	4	0	19	18	3	8	0	19	-20	-14	1	1	10	-8
6	0	0	33	-33	15	2	0	7	-8	1	5	0	9	10	4	8	0	35	-36	-13	1	1	19	-18
8	0	0	47	-47	16	2	0	39	40	2	5	0	84	-86	5	8	0	36	35	-11	1	1	37	37
10	0	0	37	36	17	2	0	9	-13	3	5	0	19	-17	12	8	0	11	-10	-10	1	1	17	16
12	0	0	14	13	18	2	0	32	-33	4	5	0	28	27	1	9	0	9	9	-9	1	1	52	-54
14	0	0	17	-18	1	3	0	49	-48	5	5	0	22	-20	2	9	0	21	-21	-8	1	1	34	34
16	0	0	46	46	2	3	0	9	-7	6	5	0	9	-9	3	9	0	16	15	-7	1	1	16	-16
18	0	0	13	-13	3	3	0	14	12	7	5	0	16	-15	4	9	0	8	-6	-6	1	1	14	14
20	0	0	8	-4	4	3	0	94	-94	9	5	0	22	21	5	9	0	25	-24	-5	1	1	93	90
3	1	0	5	3	7	3	0	44	44	13	5	0	27	-26	6	9	0	19	19	-4	1	1	53	-53
4	1	0	82	82	8	3	0	56	56	17	5	0	15	16	8	9	0	7	6	-3	1	1	31	-31
5	1	0	98	-100	9	3	0	22	24	0	6	0	35	-33	9	9	0	21	20	-2	1	1	36	-38
6	1	0	87	-85	10	3	0	29	29	1	6	0	21	-20	10	9	0	14	-14	1	1	1	43	45
7	1	0	24	25	12	3	0	12	-13	2	6	0	39	38	4	10	0	16	-17	2	1	1	42	-42
8	1	0	20	20	13	3	0	31	-30	4	6	0	7	6	-19	0	1	34	-36	3	1	1	86	-90
9	1	0	61	61	14	3	0	9	9	5	6	0	21	22	-17	0	1	22	23	4	1	1	46	-48
10	1	0	49	49	15	3	0	13	-15	7	6	0	19	19	-15	0	1	36	35	5	1	1	56	58
11	1	0	17	-19	16	3	0	17	-16	8	6	0	49	49	-13	0	1	24	-25	6	1	1	12	11
12	1	0	30	-29	18	3	0	12	-15	9	6	0	36	-36	-11	0	1	9	-9	8	1	1	48	47
16	1	0	20	21	0	4	0	72	72	11	6	0	24	-24	-9	0	1	92	91	10	1	1	9	10
19	1	0	8	8	1	4	0	19	-17	12	6	0	16	-16	-7	0	1	136	-131	12	1	1	56	-57
0	2	0	10	-12	2	4	0	35	-34	13	6	0	19	19	-5	0	1	89	-88	15	1	1	19	-23
2	2	0	51	-53	3	4	0	61	-60	15	6	0	9	9	-3	0	1	58	-58	16	1	1	34	33
3	2	0	50	50	4	4	0	39	-40	16	6	0	11	10	3	0	1	87	-91	18	1	1	7	-9
4	2	0	74	-76	5	4	0	7	7	1	7	0	10	10	5	0	1	177	-184	-18	2	1	7	-8
5	2	0	10	-8	6	4	0	47	-44	2	7	0	17	-16	7	0	1	78	79	-17	2	1	16	16
6	2	0	27	-30	7	4	0	16	18	3	7	0	16	-15	9	0	1	12	11	-16	2	1	8	-5
7	2	0	41	40	8	4	0	21	22	6	7	0	39	40	11	0	1	10	-11	-15	2	1	9	-9
8	2	0	71	70	9	4	0	10	-10	7	7	0	36	36	13	0	1	41	41	-9	2	1	6	-6
9	2	0	28	28	10	4	0	6	6	9	7	0	7	-5	15	0	1	46	49	-8	2	1	7	6
10	2	0	7	-6	12	4	0	6	-6	10	7	0	34	-34	17	0	1	54	-55	-7	2	1	30	-27
11	2	0	32	-31	13	4	0	8	10	14	7	0	22	22	19	0	1	10	-9	-6	2	1	9	8
12	2	0	23	-22	14	4	0	32	-31	0	8	0	37	37	-18	1	1	16	-16	-5	2	1	19	-24
13	2	0	12	-13	15	4	0	9	8	1	8	0	23	-23	-17	1	1	14	13	-4	2	1	92	89

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	3	8	18	-17	-4	4	8	34	32	-4	5	8	11	9	3	0	9	26	26	-4	2	9	11	-11
2	3	8	26	27	-3	4	8	20	-20	-2	5	8	9	-6	0	1	9	16	-16	-3	2	9	10	-10
4	3	8	9	9	-2	4	8	19	-21	0	5	8	13	14	1	1	9	37	38	-1	2	9	9	-10
7	3	8	10	-9	0	4	8	19	-17	1	5	8	21	-19	2	1	9	12	-10	2	2	9	19	18
-9	4	8	10	-11	1	4	8	15	14	2	5	8	15	16	3	1	9	22	21	-3	3	9	18	-16
-8	4	8	11	-10	2	4	8	20	20	-3	0	9	21	21	4	1	9	20	21	-2	3	9	8	-9
-7	4	8	8	11	3	4	8	12	9	1	0	9	29	-28	-5	2	9	27	28	0	3	9	11	-13
-6	4	8	8	-9	-6	5	8	9	10															

APPENDIX FIVE

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	0	10	-12	11	3	0	11	10	10	1	1	75	-78	5	3	1	30	-31	12	5	1	15	-12
6	0	0	140	147	13	3	0	51	-53	11	1	1	44	44	6	3	1	41	-41	13	5	1	17	15
8	0	0	79	-81	15	3	0	23	23	12	1	1	15	13	7	3	1	16	-16	0	6	1	12	-11
10	0	0	38	-38	19	3	0	16	-13	13	1	1	7	8	9	3	1	26	24	2	6	1	18	16
12	0	0	15	15	21	3	0	28	28	14	1	1	18	-19	10	3	1	30	31	4	6	1	11	-8
14	0	0	59	-57	0	4	0	69	71	15	1	1	13	14	14	3	1	10	9	6	6	1	15	12
16	0	0	59	62	2	4	0	27	-28	16	1	1	12	-12	15	3	1	7	-6	8	6	1	9	-5
18	0	0	75	-74	4	4	0	10	-11	17	1	1	8	-7	18	3	1	12	-14	9	6	1	12	-9
20	0	0	36	-35	6	4	0	20	21	18	1	1	13	8	19	3	1	18	17	10	6	1	12	-10
22	0	0	18	-19	8	4	0	17	-18	19	1	1	12	-8	21	3	1	8	-14	11	6	1	15	-13
3	1	0	120	126	10	4	0	20	-20	20	1	1	9	3	0	4	1	90	93	12	6	1	13	13
7	1	0	33	34	12	4	0	20	20	21	1	1	53	51	1	4	1	16	15	1	7	1	9	8
9	1	0	7	7	14	4	0	13	-14	22	1	1	18	15	2	4	1	27	-28	0	0	2	190	-168
11	1	0	42	-42	1	5	0	14	12	23	1	1	13	-14	4	4	1	31	32	1	0	2	168	-153
13	1	0	21	21	3	5	0	14	15	0	2	1	53	-54	8	4	1	15	-14	2	0	2	138	131
15	1	0	10	-11	5	5	0	28	-29	2	2	1	27	27	10	4	1	24	25	3	0	2	18	-19
17	1	0	21	-20	7	5	0	41	43	3	2	1	14	14	11	4	1	12	9	4	0	2	67	66
19	1	0	20	19	9	5	0	9	9	4	2	1	45	-44	12	4	1	14	-13	5	0	2	30	31
21	1	0	10	-6	11	5	0	49	-49	6	2	1	44	41	13	4	1	8	-2	6	0	2	16	-18
23	1	0	11	-12	15	5	0	14	-14	7	2	1	23	23	14	4	1	41	-42	7	0	2	16	-17
0	2	0	208	-210	2	6	0	17	-15	8	2	1	39	40	15	4	1	12	-11	8	0	2	35	-35
2	2	0	144	143	4	6	0	8	3	9	2	1	36	36	16	4	1	39	39	9	0	2	94	97
4	2	0	33	32	6	6	0	8	5	10	2	1	21	-22	17	4	1	8	5	10	0	2	16	16
6	2	0	37	-36	8	6	0	29	-25	11	2	1	15	15	18	4	1	25	-23	12	0	2	31	-32
10	2	0	39	38	10	6	0	12	10	14	2	1	13	15	2	5	1	7	-4	13	0	2	39	-39
12	2	0	22	22	1	1	1	43	-44	16	2	1	34	-34	3	5	1	14	11	14	0	2	29	31
14	2	0	12	15	2	1	1	19	18	17	2	1	12	-8	4	5	1	7	-2	15	0	2	49	49
16	2	0	49	-48	3	1	1	196	-197	18	2	1	26	27	5	5	1	16	-14	16	0	2	45	-46
18	2	0	26	27	4	1	1	69	-66	19	2	1	9	7	6	5	1	11	-6	17	0	2	26	-26
20	2	0	31	-29	5	1	1	103	102	20	2	1	13	-12	7	5	1	14	12	18	0	2	39	39
1	3	0	56	-57	6	1	1	115	114	1	3	1	48	-50	8	5	1	15	-13	19	0	2	9	-7
5	3	0	46	45	7	1	1	13	13	2	3	1	40	-42	9	5	1	9	7	20	0	2	10	-6
7	3	0	27	-26	8	1	1	24	-24	3	3	1	88	88	10	5	1	17	-14	21	0	2	36	34
9	3	0	37	40	9	1	1	38	-39	4	3	1	11	9	11	5	1	18	-16	23	0	2	21	22

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	1	2	48	47	20	2	2	22	22	5	5	2	33	35	19	1	3	20	20	8	4	3	20	21
2	1	2	87	86	2	3	2	28	27	6	5	2	22	22	20	1	3	19	17	9	4	3	11	10
3	1	2	46	-48	4	3	2	32	32	7	5	2	31	-30	21	1	3	13	-15	10	4	3	20	-19
4	1	2	24	26	5	3	2	41	-42	8	5	2	15	-11	22	1	3	18	-20	11	4	3	16	-14
5	1	2	49	50	6	3	2	11	-9	9	5	2	17	-16	0	2	3	16	17	12	4	3	18	-18
6	1	2	69	70	7	3	2	36	37	10	5	2	32	-33	1	2	3	36	34	14	4	3	22	22
7	1	2	12	-13	8	3	2	7	7	11	5	2	29	29	2	2	3	52	-54	15	4	3	26	26
8	1	2	55	56	9	3	2	8	-11	12	5	2	7	-1	4	2	3	25	26	16	4	3	28	-30
9	1	2	14	-15	10	3	2	11	-11	13	5	2	17	-14	5	2	3	14	15	17	4	3	17	-15
10	1	2	32	33	11	3	2	18	-18	0	6	2	18	-16	7	2	3	13	12	1	5	3	10	7
11	1	2	28	28	12	3	2	10	-10	1	6	2	14	-9	9	2	3	42	-43	2	5	3	17	18
12	1	2	52	55	13	3	2	36	35	2	6	2	14	13	10	2	3	16	18	3	5	3	10	-8
13	1	2	24	-25	14	3	2	26	22	3	6	2	7	7	11	2	3	13	-13	4	5	3	18	-16
14	1	2	15	19	15	3	2	18	-18	4	6	2	9	-3	12	2	3	9	10	5	5	3	16	16
16	1	2	12	14	16	3	2	7	-7	5	6	2	9	8	14	2	3	24	-24	6	5	3	15	13
17	1	2	14	13	18	3	2	8	6	6	6	2	11	-10	16	2	3	17	19	7	5	3	8	1
18	1	2	16	14	19	3	2	12	11	8	6	2	20	19	17	2	3	19	17	8	5	3	11	11
19	1	2	15	-15	21	3	2	17	-18	9	6	2	21	20	18	2	3	13	-13	10	5	3	7	2
20	1	2	10	-9	0	4	2	19	-17	1	1	3	105	-97	19	2	3	15	-13	11	5	3	14	14
0	2	2	28	29	1	4	2	9	-4	2	1	3	102	91	1	3	3	14	15	12	5	3	17	17
2	2	2	46	-46	2	4	2	15	12	3	1	3	61	-57	2	3	3	16	15	13	5	3	9	-5
3	2	2	58	-57	3	4	2	16	16	4	1	3	54	54	3	3	3	10	-8	0	6	3	19	17
4	2	2	12	12	6	4	2	9	-7	5	1	3	41	-41	6	3	3	17	17	1	6	3	17	13
7	2	2	41	43	7	4	2	11	-12	6	1	3	42	-43	7	3	3	10	-11	2	6	3	14	-11
8	2	2	63	64	8	4	2	11	-13	7	1	3	42	43	10	3	3	19	-19	4	6	3	14	13
9	2	2	24	23	9	4	2	12	13	8	1	3	25	25	11	3	3	8	-8	8	6	3	8	-4
10	2	2	14	-14	10	4	2	12	12	9	1	3	21	21	12	3	3	16	14	11	6	3	17	15
11	2	2	67	67	12	4	2	16	-14	10	1	3	60	61	15	3	3	12	10	0	0	4	156	-142
13	2	2	40	41	13	4	2	29	-28	11	1	3	21	21	18	3	3	17	17	1	0	4	169	159
14	2	2	9	-11	14	4	2	11	7	12	1	3	34	-34	0	4	3	22	-22	2	0	4	254	-231
15	2	2	22	-22	1	5	2	8	6	13	1	3	14	16	1	4	3	53	-56	3	0	4	79	75
16	2	2	18	19	2	5	2	7	5	15	1	3	15	-17	2	4	3	59	62	4	0	4	8	-9
17	2	2	10	5	3	5	2	15	-16	16	1	3	10	8	5	4	3	14	15	5	0	4	33	33
18	2	2	34	-35	4	5	2	9	-9	17	1	3	14	14	7	4	3	20	-21	6	0	4	25	-25

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
7	0	4	44	43	9	2	4	16	15	1	5	4	11	-10	20	1	5	30	-26	8	4	5	13	-11
9	0	4	41	-41	11	2	4	23	-23	2	5	4	19	-18	0	2	5	12	-12	9	4	5	17	-17
11	0	4	23	24	12	2	4	10	-9	3	5	4	13	8	1	2	5	46	-46	12	4	5	14	13
12	0	4	13	13	13	2	4	21	21	4	5	4	32	31	2	2	5	11	9	13	4	5	13	13
15	0	4	49	-49	14	2	4	7	-4	5	5	4	23	-23	4	2	5	22	-22	15	4	5	20	-21
16	0	4	36	36	15	2	4	30	29	6	5	4	27	-28	6	2	5	8	-7	1	5	5	7	2
17	0	4	54	53	16	2	4	11	-12	9	5	4	8	3	7	2	5	42	-45	2	5	5	12	-11
18	0	4	10	5	17	2	4	23	-21	10	5	4	33	33	8	2	5	15	-15	4	5	5	18	19
19	0	4	13	12	18	2	4	27	27	11	5	4	9	-1	11	2	5	12	-13	5	5	5	9	6
20	0	4	53	52	19	2	4	13	9	12	5	4	18	-13	13	2	5	14	-15	6	5	5	7	1
21	0	4	9	-7	20	2	4	11	-14	13	5	4	26	27	14	2	5	17	17	7	5	5	10	3
22	0	4	12	12	1	3	4	20	21	15	5	4	8	11	15	2	5	12	9	8	5	5	9	6
1	1	4	27	-28	4	3	4	48	-49	0	6	4	8	6	17	2	5	17	-16	9	5	5	8	1
2	1	4	49	-49	5	3	4	11	10	1	6	4	16	16	19	2	5	10	13	10	5	5	18	18
3	1	4	19	-18	6	3	4	13	16	2	6	4	10	-2	2	3	5	8	-6	1	6	5	21	-18
4	1	4	69	70	7	3	4	30	-30	3	6	4	8	-7	3	3	5	10	11	4	6	5	9	-13
5	1	4	48	-50	11	3	4	22	23	6	6	4	8	8	4	3	5	26	26	5	6	5	12	12
6	1	4	7	7	12	3	4	21	20	7	6	4	15	17	5	3	5	17	-19	8	6	5	9	10
9	1	4	6	6	13	3	4	8	-6	9	6	4	11	-12	6	3	5	12	-11	0	0	6	20	-18
10	1	4	31	31	14	3	4	18	-19	1	1	5	45	42	7	3	5	15	-15	1	0	6	50	-48
11	1	4	10	-11	16	3	4	10	8	2	1	5	12	-11	8	3	5	9	9	2	0	6	18	-17
12	1	4	16	-16	17	3	4	8	5	3	1	5	26	-27	9	3	5	11	-10	3	0	6	58	58
13	1	4	10	12	20	3	4	15	-15	4	1	5	48	-48	12	3	5	19	-18	4	0	6	55	-55
14	1	4	12	12	0	4	4	12	12	5	1	5	7	2	14	3	5	10	10	5	0	6	36	35
15	1	4	7	7	1	4	4	8	-2	6	1	5	57	56	15	3	5	8	9	6	0	6	24	-24
18	1	4	13	-13	2	4	4	14	-14	7	1	5	24	25	16	3	5	9	-2	7	0	6	20	-18
20	1	4	9	9	4	4	4	8	3	8	1	5	23	-21	0	4	5	14	-13	8	0	6	11	-12
0	2	4	62	-60	5	4	4	10	9	9	1	5	18	19	1	4	5	57	59	9	0	6	17	17
1	2	4	12	-9	7	4	4	8	5	11	1	5	12	-11	2	4	5	20	-20	10	0	6	48	50
2	2	4	19	19	8	4	4	9	7	12	1	5	41	43	3	4	5	11	-12	11	0	6	33	-33
4	2	4	42	-41	9	4	4	12	-10	14	1	5	21	-21	4	4	5	18	17	12	0	6	21	-21
5	2	4	31	-32	11	4	4	10	-9	15	1	5	9	-4	5	4	5	30	-28	13	0	6	19	-19
6	2	4	24	22	14	4	4	8	2	16	1	5	15	16	6	4	5	16	-16	14	0	6	12	11
7	2	4	11	12	15	4	4	10	-6	19	1	5	18	-19	7	4	5	29	29	15	0	6	32	32

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
16	0	6	18	16	6	3	6	28	-29	8	1	7	7	-7	6	4	7	13	13	13	1	8	9	-8
17	0	6	26	-24	7	3	6	12	12	9	1	7	8	-3	7	4	7	13	-11	0	2	8	8	2
20	0	6	14	-17	8	3	6	7	1	10	1	7	16	13	8	4	7	14	-12	1	2	8	12	-13
1	1	6	32	-34	9	3	6	7	-5	11	1	7	15	12	9	4	7	17	16	2	2	8	18	17
2	1	6	20	20	10	3	6	15	12	12	1	7	23	-21	10	4	7	8	10	3	2	8	14	13
3	1	6	9	7	12	3	6	14	-12	13	1	7	12	-12	2	5	7	8	5	4	2	8	7	-1
4	1	6	56	-59	13	3	6	8	-6	14	1	7	15	15	3	5	7	8	8	5	2	8	12	-10
5	1	6	12	-14	14	3	6	15	15	15	1	7	10	-8	4	5	7	10	-7	6	2	8	9	-6
6	1	6	10	-10	16	3	6	10	-13	17	1	7	15	-18	0	0	8	22	20	7	2	8	17	-15
7	1	6	11	-12	0	4	6	16	-16	18	1	7	18	-20	2	0	8	38	38	8	2	8	14	-11
8	1	6	14	-14	1	4	6	9	-5	0	2	7	8	-10	3	0	8	67	-68	9	2	8	21	21
10	1	6	37	-40	2	4	6	9	7	1	2	7	18	19	4	0	8	47	44	10	2	8	10	9
13	1	6	9	9	3	4	6	9	4	3	2	7	20	-22	5	0	8	22	-21	12	2	8	8	-4
14	1	6	22	-22	4	4	6	12	9	5	2	7	18	-17	6	0	8	7	3	13	2	8	15	-15
16	1	6	13	-14	6	4	6	8	3	6	2	7	10	-10	7	0	8	25	24	2	3	8	18	16
0	2	6	45	45	7	4	6	9	-6	8	2	7	23	25	8	0	8	27	26	3	3	8	11	9
1	2	6	11	13	10	4	6	13	11	9	2	7	7	-2	9	0	8	36	-33	5	3	8	10	-10
2	2	6	6	-7	1	5	6	9	-4	10	2	7	9	-8	10	0	8	16	-15	6	3	8	11	10
3	2	6	19	-20	2	5	6	22	21	11	2	7	14	12	11	0	8	7	1	7	3	8	8	9
4	2	6	11	-11	3	5	6	8	1	12	2	7	7	-4	13	0	8	21	20	10	3	8	9	-4
6	2	6	11	-12	4	5	6	30	-29	15	2	7	11	-9	14	0	8	9	1	11	3	8	10	-8
7	2	6	26	-25	5	5	6	11	-8	3	3	7	7	-6	15	0	8	30	-29	0	4	8	9	6
8	2	6	14	-14	6	5	6	20	19	4	3	7	12	-12	16	0	8	31	-33	1	4	8	9	6
9	2	6	35	-35	7	5	6	10	10	5	3	7	7	3	1	1	8	13	13	4	5	8	13	14
10	2	6	31	-31	8	5	6	14	13	6	3	7	13	11	2	1	8	10	-9	5	5	8	16	18
11	2	6	7	3	9	5	6	20	19	7	3	7	9	8	3	1	8	13	-13	1	1	9	11	-7
12	2	6	29	28	12	5	6	16	15	10	3	7	13	-13	4	1	8	7	1	2	1	9	14	13
13	2	6	11	-10	1	6	6	8	-11	12	3	7	8	10	5	1	8	17	19	3	1	9	17	15
15	2	6	13	-13	2	1	7	36	-37	0	4	7	15	15	6	1	8	21	-20	4	1	9	21	-19
17	2	6	18	19	3	1	7	53	54	1	4	7	25	-25	7	1	8	10	7	5	1	9	23	-24
19	2	6	10	-13	4	1	7	17	-15	2	4	7	18	-20	8	1	8	15	-15	6	1	9	9	5
2	3	6	36	-37	5	1	7	32	33	3	4	7	15	17	10	1	8	7	-2	7	1	9	7	6
4	3	6	13	14	6	1	7	43	-44	4	4	7	23	-25	11	1	8	11	8	8	1	9	17	-18
5	3	6	15	17	7	1	7	23	-24	5	4	7	15	13	12	1	8	11	-10	9	1	9	9	10

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
10	1	9	10	-6	5	2	9	11	10	2	4	9	10	11	7	0	10	21	-17	2	2	10	11	10
11	1	9	17	-17	6	2	9	14	15	0	0	10	33	35	8	0	10	15	-13	4	2	10	9	7
12	1	9	17	17	9	2	9	8	3	1	0	10	7	7	9	0	10	11	6	5	2	10	16	18
13	1	9	13	13	10	2	9	10	9	2	0	10	12	-14	10	0	10	10	14	7	2	10	12	9
14	1	9	10	-14	3	3	9	12	-10	3	0	10	10	6	3	1	10	15	15	8	2	10	12	15
0	2	9	10	8	4	3	9	12	11	4	0	10	10	10	6	1	10	12	11	3	1	11	21	-22
3	2	9	7	8	5	3	9	15	15	5	0	10	29	-32	7	1	10	8	7	6	1	11	11	18
4	2	9	8	8	6	3	9	14	-12	6	0	10	41	42	0	2	10	16	-18					

APPENDIX SIX

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	0	77	-73	14	2	0	28	-28	3	5	0	4	4	-9	0	1	64	-57	10	1	1	6	7
6	0	0	29	-27	16	2	0	7	6	4	5	0	17	-17	-7	0	1	11	-8	12	1	1	13	13
8	0	0	22	20	1	3	0	12	14	5	5	0	38	37	-5	0	1	88	85	13	1	1	15	15
10	0	0	34	31	2	3	0	45	43	6	5	0	11	12	-3	0	1	29	27	16	1	1	4	-5
12	0	0	24	23	3	3	0	26	22	7	5	0	24	24	3	0	1	103	106	18	1	1	4	-3
14	0	0	8	8	4	3	0	67	64	9	5	0	4	5	5	0	1	82	81	-18	2	1	5	-5
16	0	0	12	-11	5	3	0	22	18	11	5	0	11	-11	7	0	1	19	-18	-17	2	1	7	-8
18	0	0	6	-5	6	3	0	10	10	12	5	0	5	-4	9	0	1	56	-51	-15	2	1	6	-6
2	1	0	25	-26	9	3	0	7	-6	13	5	0	11	-12	11	0	1	38	-36	-14	2	1	18	17
3	1	0	43	-42	10	3	0	9	7	14	5	0	7	-7	13	0	1	21	20	-13	2	1	10	11
4	1	0	74	-73	11	3	0	16	17	15	5	0	5	-7	15	0	1	20	-19	-12	2	1	4	-4
5	1	0	5	5	12	3	0	19	18	0	6	0	13	-12	17	0	1	17	17	-11	2	1	13	15
6	1	0	79	74	13	3	0	18	18	1	6	0	19	18	-18	1	1	5	6	-10	2	1	22	-21
7	1	0	20	22	14	3	0	19	20	2	6	0	14	-14	-17	1	1	9	-8	-8	2	1	23	21
8	1	0	39	36	15	3	0	9	-9	3	6	0	25	24	-16	1	1	18	18	-7	2	1	27	-29
11	1	0	12	-13	16	3	0	9	8	4	6	0	20	-20	-15	1	1	14	-13	-6	2	1	67	65
13	1	0	5	6	0	4	0	39	34	5	6	0	27	28	-12	1	1	16	16	-4	2	1	106	109
14	1	0	6	-8	1	4	0	24	-24	6	6	0	19	-21	-11	1	1	4	5	-3	2	1	51	50
15	1	0	4	3	2	4	0	18	-18	7	6	0	28	-26	-10	1	1	15	15	-2	2	1	11	11
17	1	0	9	9	3	4	0	18	-19	8	6	0	5	-3	-9	1	1	13	14	-1	2	1	15	17
18	1	0	5	-6	4	4	0	4	4	9	6	0	7	-7	-8	1	1	5	3	0	2	1	7	7
0	2	0	96	-92	5	4	0	44	-41	10	6	0	11	-10	-7	1	1	14	12	1	2	1	108	109
1	2	0	85	-81	6	4	0	8	8	11	6	0	9	8	-6	1	1	35	33	2	2	1	26	-26
2	2	0	8	10	7	4	0	7	-6	13	6	0	9	7	-5	1	1	58	-63	3	2	1	4	5
3	2	0	151	-146	8	4	0	8	-8	1	7	0	16	14	-2	1	1	59	-59	4	2	1	28	-22
4	2	0	10	-9	9	4	0	27	26	2	7	0	6	-8	-1	1	1	22	-21	5	2	1	18	-19
5	2	0	23	20	10	4	0	12	12	10	N7	0	5	-5	1	1	1	46	49	7	2	1	4	4
6	2	0	36	-32	11	4	0	11	-12	0	8	0	6	4	2	1	1	93	-93	8	2	1	21	-19
7	2	0	43	-43	12	4	0	12	12	2	8	0	13	13	3	1	1	28	26	9	2	1	10	10
8	2	0	21	-18	13	4	0	8	-10	3	8	0	8	-7	4	1	1	22	-22	10	2	1	15	13
9	2	0	26	-28	14	4	0	8	8	-17	0	1	8	-9	5	1	1	6	-7	11	2	1	18	18
10	2	0	26	-25	15	4	0	13	-13	-15	0	1	26	-26	6	1	1	25	-25	12	2	1	16	-16
11	2	0	14	-13	16	4	0	4	5	-13	0	1	9	-9	7	1	1	67	-62	13	2	1	17	17
12	2	0	14	-15	1	5	0	20	-20	-11	0	1	33	-32	9	1	1	12	10	14	2	1	12	-11

OBSERVED AND CALCULATED STRUCTURE FACTORS :

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
15	2	1	14	13	-9	4	1	12	11	2	5	1	17	17	-3	7	1	9	7	12	0	2	6	6
16	2	1	5	-6	-8	4	1	23	23	4	5	1	14	-13	-2	7	1	9	-7	14	0	2	13	13
-17	3	1	5	6	-7	4	1	46	45	5	5	1	7	6	-1	7	1	11	9	16	0	2	16	16
-14	3	1	9	-11	-6	4	1	16	17	6	5	1	4	6	0	7	1	25	-23	-17	1	2	5	4
-13	3	1	8	-9	-5	4	1	8	8	7	5	1	18	-18	1	7	1	14	14	-16	1	2	4	3
-12	3	1	17	-16	-4	4	1	25	-26	8	5	1	49	50	2	7	1	7	-7	-15	1	2	7	7
-11	3	1	9	-10	-3	4	1	44	-43	10	5	1	27	27	3	7	1	12	14	-14	1	2	11	11
-10	3	1	22	-23	-1	4	1	48	-45	11	5	1	11	-11	4	7	1	5	1	-13	1	2	10	11
-9	3	1	26	-26	0	4	1	15	16	12	5	1	13	13	5	7	1	20	20	-12	1	2	21	-21
-8	3	1	24	22	1	4	1	25	-22	-13	6	1	5	3	6	7	1	12	12	-11	1	2	6	6
-7	3	1	12	-12	2	4	1	26	25	-12	6	1	7	-7	9	7	1	5	-6	-10	1	2	12	-12
-6	3	1	26	-25	3	4	1	4	-3	-11	6	1	9	8	10	7	1	5	-4	-8	1	2	45	-44
-5	3	1	37	-36	5	4	1	6	-6	-10	6	1	9	-10	-6	8	1	5	-5	-7	1	2	47	46
-4	3	1	20	-18	6	4	1	15	14	-8	6	1	12	-11	-4	8	1	16	-13	-6	1	2	24	-22
-3	3	1	34	30	7	4	1	24	-25	-7	6	1	11	-11	-3	8	1	7	6	-5	1	2	15	15
-2	3	1	20	-20	8	4	1	14	-14	-6	6	1	24	-22	-2	8	1	10	-8	-4	1	2	46	-42
-1	3	1	37	-37	9	4	1	41	-41	-4	6	1	7	6	-1	8	1	5	6	-3	1	2	8	-8
0	3	1	42	39	10	4	1	28	-28	-3	6	1	9	-8	1	8	1	6	6	-2	1	2	25	-25
1	3	1	12	-13	11	4	1	20	-20	-2	6	1	5	3	2	8	1	8	6	-1	1	2	12	-14
2	3	1	37	-34	12	4	1	9	10	0	6	1	12	-11	6	8	1	6	-4	0	1	2	73	-79
3	3	1	14	15	14	4	1	4	5	1	6	1	6	-6	-18	0	2	9	-9	1	1	2	36	34
4	3	1	15	-12	15	4	1	9	-10	2	6	1	34	-31	-16	0	2	10	11	3	1	2	51	-50
6	3	1	42	41	-12	5	1	12	13	4	6	1	20	-20	-14	0	2	15	-14	4	1	2	4	-3
7	3	1	9	9	-10	5	1	10	9	5	6	1	6	8	-12	0	2	10	10	5	1	2	53	-51
8	3	1	23	20	-9	5	1	12	-12	6	6	1	8	7	-10	0	2	17	-17	6	1	2	48	46
9	3	1	38	37	-8	5	1	32	-33	7	6	1	24	-24	-8	0	2	43	-45	7	1	2	9	8
11	3	1	29	28	-7	5	1	13	13	9	6	1	10	-12	-6	0	2	20	-23	8	1	2	7	7
12	3	1	20	-19	-6	5	1	9	-8	10	6	1	19	21	-4	0	2	28	27	9	1	2	21	-19
14	3	1	14	-14	-5	5	1	17	-17	-10	7	1	11	-10	-2	0	2	17	20	10	1	2	11	-11
16	3	1	7	8	-3	5	1	5	-5	-8	7	1	11	10	2	0	2	66	-71	11	1	2	8	-8
-16	4	1	6	-7	-2	5	1	37	36	-7	7	1	13	14	4	0	2	27	-28	13	1	2	8	9
-14	4	1	6	-5	-1	5	1	12	11	-6	7	1	5	3	6	0	2	18	19	14	1	2	22	-23
-12	4	1	15	15	0	5	1	51	48	-5	7	1	12	12	8	0	2	51	47	15	1	2	21	-22
-10	4	1	29	28	1	5	1	8	-6	-4	7	1	6	4	10	0	2	16	15	16	1	2	7	-9

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
17	1	2	8	-9	-17	3	2	5	-3	-8	4	2	21	20	1	5	2	15	-15	4	7	2	19	18
18	1	2	9	13	-15	3	2	7	7	-7	4	2	23	22	3	5	2	18	19	6	7	2	13	14
-17	2	2	5	-6	-14	3	2	4	1	-6	4	2	10	10	4	5	2	16	-16	7	7	2	10	-11
-16	2	2	4	-4	-13	3	2	26	27	-5	4	2	15	15	5	5	2	16	14	8	7	2	5	5
-15	2	2	11	-11	-12	3	2	16	16	-2	4	2	15	17	6	5	2	10	10	9	7	2	14	14
-14	2	2	30	-31	-11	3	2	6	7	-1	4	2	28	-27	7	5	2	30	30	-5	8	2	4	-3
-13	2	2	11	-12	-10	3	2	32	31	0	4	2	34	32	8	5	2	16	-17	-4	8	2	6	-4
-12	2	2	28	-28	-9	3	2	27	-26	1	4	2	8	-9	9	5	2	12	-12	-3	8	2	5	2
-11	2	2	11	11	-8	3	2	20	20	2	4	2	21	-24	10	5	2	4	4	-1	8	2	7	-7
-9	2	2	8	9	-7	3	2	76	-77	4	4	2	28	-25	13	5	2	8	8	0	8	2	11	-11
-8	2	2	33	33	-6	3	2	30	-31	5	4	2	14	14	-11	6	2	17	18	1	8	2	14	-11
-7	2	2	52	49	-4	3	2	22	-24	6	4	2	18	-16	-10	6	2	5	5	2	8	2	6	-4
-6	2	2	32	32	-3	3	2	44	47	7	4	2	17	17	-8	6	2	18	17	3	8	2	21	-22
-5	2	2	13	9	-2	3	2	6	5	8	4	2	16	-16	-7	6	2	6	6	-15	0	3	18	-18
-4	2	2	18	17	0	3	2	6	-5	9	4	2	21	20	-5	6	2	33	-31	-13	0	3	21	-20
-3	2	2	8	-8	1	3	2	39	37	10	4	2	7	-9	-4	6	2	4	-4	-11	0	3	40	-37
-2	2	2	83	-84	2	3	2	13	13	11	4	2	5	5	-3	6	2	23	-22	-9	0	3	9	-7
-1	2	2	25	29	3	3	2	27	24	13	4	2	12	-12	0	6	2	14	13	-7	0	3	4	2
0	2	2	33	-34	4	3	2	24	-24	14	4	2	9	-9	2	6	2	13	14	-5	0	3	69	-68
1	2	2	57	60	5	3	2	36	35	15	4	2	9	-8	4	6	2	10	-10	-3	0	3	41	-46
2	2	2	17	-13	7	3	2	32	-29	16	4	2	7	-7	5	6	2	14	-12	1	0	3	42	-40
3	2	2	57	55	8	3	2	16	-14	-13	5	2	13	-14	7	6	2	25	-24	3	0	3	73	-65
4	2	2	20	22	9	3	2	21	20	-12	5	2	8	-7	10	6	2	12	-11	5	0	3	10	11
5	2	2	33	31	11	3	2	21	20	-11	5	2	10	-10	12	6	2	8	-8	7	0	3	11	-10
6	2	2	20	20	12	3	2	5	-7	-10	5	2	23	-23	-7	7	2	12	-10	9	0	3	42	-40
7	2	2	9	-7	13	3	2	13	13	-9	5	2	28	-28	-6	7	2	13	13	11	0	3	14	-14
8	2	2	4	4	14	3	2	6	6	-8	5	2	19	-20	-4	7	2	12	12	13	0	3	14	14
9	2	2	6	-6	16	3	2	9	9	-6	5	2	11	10	-3	7	2	8	7	15	0	3	7	5
10	2	2	12	-11	-16	4	2	5	3	-5	5	2	7	-8	-2	7	2	20	20	17	0	3	14	13
12	2	2	14	-14	-13	4	2	4	2	-4	5	2	14	15	-1	7	2	22	20	-17	1	3	9	-8
13	2	2	21	21	-12	4	2	18	18	-3	5	2	28	-26	0	7	2	14	15	-15	1	3	4	-3
15	2	2	5	4	-11	4	2	12	-11	-2	5	2	5	-4	1	7	2	5	6	-14	1	3	12	-12
16	2	2	10	11	-10	4	2	28	28	-1	5	2	40	-38	2	7	2	7	7	-13	1	3	30	30
17	2	2	8	-9	-9	4	2	14	-15	0	5	2	4	1	3	7	2	9	-7	-12	1	3	30	30

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-11	1	3	40	41	-9	2	3	11	-10	-5	3	3	34	35	-3	4	3	4	1	10	5	3	10	10
-10	1	3	28	28	-8	2	3	30	-30	-4	3	3	71	-71	-2	4	3	17	-16	11	5	3	8	7
-9	1	3	4	4	-7	2	3	17	14	-3	3	3	11	11	-1	4	3	35	-36	12	5	3	17	17
-8	1	3	18	-16	-6	2	3	9	10	-2	3	3	56	-59	0	4	3	23	24	13	5	3	11	12
-7	1	3	44	39	-5	2	3	75	76	-1	3	3	59	-62	1	4	3	11	13	14	5	3	7	7
-6	1	3	10	-9	-3	2	3	55	56	0	3	3	43	41	2	4	3	18	18	-11	6	3	8	9
-5	1	3	21	-22	-2	2	3	6	4	1	3	3	58	-62	3	4	3	15	-14	-10	6	3	4	-4
-4	1	3	31	32	-1	2	3	30	30	2	3	3	18	20	4	4	3	26	24	-9	6	3	4	-3
-3	1	3	37	-37	0	2	3	24	-26	3	3	3	57	-57	5	4	3	17	-18	-7	6	3	9	9
-2	1	3	56	60	1	2	3	32	-32	4	3	3	34	33	6	4	3	19	20	-6	6	3	15	14
-1	1	3	54	59	2	2	3	46	45	5	3	3	14	-13	9	4	3	11	-10	-5	6	3	31	30
0	1	3	104	109	3	2	3	15	-16	6	3	3	8	7	10	4	3	16	17	-4	6	3	4	5
1	1	3	24	-24	4	2	3	14	16	7	3	3	22	-23	11	4	3	11	-12	-3	6	3	18	19
2	1	3	17	18	5	2	3	5	5	8	3	3	12	-11	12	4	3	11	11	-1	6	3	9	11
3	1	3	33	32	6	2	3	44	42	9	3	3	16	-18	13	4	3	12	-11	0	6	3	30	-30
4	1	3	46	-40	7	2	3	9	9	10	3	3	28	-28	14	4	3	10	11	1	6	3	13	11
7	1	3	19	16	8	2	3	6	6	11	3	3	29	-29	15	4	3	8	-9	2	6	3	29	-29
8	1	3	24	20	9	2	3	34	33	12	3	3	15	-15	-14	5	3	4	4	3	6	3	16	15
9	1	3	8	-8	10	2	3	24	24	13	3	3	24	-25	-12	5	3	5	6	4	6	3	23	-25
10	1	3	7	5	11	2	3	22	22	14	3	3	12	12	-10	5	3	6	-7	5	6	3	17	19
11	1	3	7	-6	12	2	3	8	9	15	3	3	6	-6	-9	5	3	9	-8	6	6	3	20	21
12	1	3	10	-11	13	2	3	26	26	16	3	3	8	8	-8	5	3	27	-25	7	6	3	4	-1
13	1	3	14	14	15	2	3	7	-8	-16	4	3	11	-11	-7	5	3	12	14	10	6	3	8	-7
14	1	3	13	-12	-17	3	3	7	6	-15	4	3	6	-6	-6	5	3	27	-26	11	6	3	6	-5
16	1	3	10	-10	-16	3	3	5	4	-13	4	3	10	-8	-5	5	3	20	-21	12	6	3	9	-10
17	1	3	8	8	-15	3	3	14	15	-12	4	3	7	8	-4	5	3	12	-13	-9	7	3	6	-4
18	1	3	5	-5	-14	3	3	8	-8	-11	4	3	12	-13	-3	5	3	21	18	-8	7	3	11	11
-17	2	3	4	-1	-13	3	3	9	8	-10	4	3	29	28	-1	5	3	43	43	-4	7	3	8	-8
-15	2	3	18	19	-12	3	3	12	-11	-9	4	3	8	8	1	5	3	14	16	-3	7	3	8	-7
-14	2	3	8	-8	-11	3	3	10	-9	-8	4	3	6	-5	3	5	3	5	5	-2	7	3	9	-8
-13	2	3	11	10	-9	3	3	13	-14	-7	4	3	27	28	4	5	3	23	-22	-1	7	3	8	5
-12	2	3	17	-18	-8	3	3	12	11	-6	4	3	27	-26	6	5	3	5	-5	0	7	3	12	-13
-11	2	3	11	10	-7	3	3	13	13	-5	4	3	19	20	7	5	3	6	-5	1	7	3	19	18
-10	2	3	26	-26	-6	3	3	37	-33	-4	4	3	17	-16	9	5	3	4	-4	3	7	3	12	13

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	7	3	7	7	-6	1	4	61	60	1	2	4	53	51	7	3	4	22	-21	-6	5	4	17	17
7	7	3	4	-5	-5	1	4	35	33	2	2	4	42	-42	8	3	4	36	-33	-5	5	4	7	-6
9	7	3	7	7	-3	1	4	4	2	3	2	4	27	27	10	3	4	26	-25	-4	5	4	10	13
-4	8	3	8	-7	-2	1	4	43	-41	4	2	4	66	-64	12	3	4	7	-7	-3	5	4	46	-45
-3	8	3	4	-5	-1	1	4	85	-84	5	2	4	9	-7	13	3	4	4	5	-1	5	4	38	-39
-2	8	3	4	-2	0	1	4	46	46	6	2	4	15	-14	14	3	4	6	6	0	5	4	22	19
1	8	3	14	-13	1	1	4	88	90	7	2	4	9	10	15	3	4	11	-12	2	5	4	9	9
5	8	3	6	-5	2	1	4	29	-28	9	2	4	9	9	16	3	4	5	-4	3	5	4	5	-5
-16	0	4	26	26	3	1	4	90	88	10	2	4	20	-20	-13	4	4	9	-8	4	5	4	14	-12
-14	0	4	19	18	4	1	4	21	-20	11	2	4	30	30	-11	4	4	23	-23	5	5	4	13	-13
-12	0	4	19	17	5	1	4	42	40	13	2	4	19	19	-9	4	4	14	-13	6	5	4	9	10
-8	0	4	11	-9	6	1	4	53	50	15	2	4	6	7	-8	4	4	30	-30	7	5	4	16	-17
-6	0	4	20	-18	7	1	4	19	18	16	2	4	7	7	-6	4	4	5	4	8	5	4	15	-17
-4	0	4	24	-23	8	1	4	15	-15	-15	3	4	13	14	-4	4	4	61	63	9	5	4	27	-27
-2	0	4	72	-76	9	1	4	12	-11	-14	3	4	4	3	-3	4	4	30	-28	10	5	4	10	11
0	0	4	23	24	10	1	4	13	-13	-13	3	4	7	6	-2	4	4	53	56	11	5	4	15	-14
2	0	4	65	-67	11	1	4	19	-18	-12	3	4	24	24	-1	4	4	25	-26	13	5	4	6	6
4	0	4	33	-37	12	1	4	16	-16	-11	3	4	4	1	0	4	4	27	28	-11	6	4	17	16
6	0	4	18	-18	13	1	4	5	5	-10	3	4	34	35	1	4	4	42	-43	-8	6	4	6	7
8	0	4	11	10	14	1	4	19	-19	-9	3	4	12	-13	2	4	4	10	-10	-7	6	4	16	18
10	0	4	46	42	-15	2	4	7	-7	-8	3	4	6	-4	3	4	4	8	-7	-6	6	4	13	-11
14	0	4	19	19	-14	2	4	15	-15	-7	3	4	18	-19	5	4	4	18	18	-4	6	4	8	9
16	0	4	12	-11	-13	2	4	7	-7	-6	3	4	8	7	6	4	4	7	7	-3	6	4	6	5
-18	1	4	7	5	-12	2	4	5	5	-5	3	4	20	-18	7	4	4	21	21	-2	6	4	15	13
-17	1	4	7	-8	-11	2	4	5	4	-3	3	4	16	17	8	4	4	32	32	0	6	4	14	14
-16	1	4	4	4	-10	2	4	17	16	-2	3	4	22	22	9	4	4	26	26	1	6	4	16	17
-15	1	4	8	-7	-9	2	4	17	-17	-1	3	4	21	-18	10	4	4	29	29	2	6	4	5	7
-14	1	4	7	7	-8	2	4	18	19	0	3	4	15	17	12	4	4	4	2	3	6	4	10	9
-13	1	4	16	-16	-7	2	4	37	-36	1	3	4	11	8	13	4	4	8	-7	4	6	4	9	-9
-12	1	4	13	-12	-5	2	4	92	-95	2	3	4	31	29	15	4	4	6	-7	5	6	4	7	-7
-11	1	4	8	-8	-4	2	4	33	-33	3	3	4	25	23	-12	5	4	8	-8	6	6	4	16	15
-10	1	4	4	-2	-2	2	4	12	-11	4	3	4	16	-18	-9	5	4	7	7	8	6	4	9	10
-8	1	4	41	41	-1	2	4	44	45	5	3	4	13	13	-8	5	4	8	-9	9	6	4	9	-9
-7	1	4	21	23	0	2	4	26	-24	6	3	4	32	-31	-7	5	4	8	8	10	6	4	4	-4

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
12	6	4	7	-6	-12	1	5	17	-17	-6	2	5	5	-4	-2	3	5	23	-27	6	4	5	3	3
-9	7	4	5	-4	-11	1	5	29	28	-5	2	5	5	4	-1	3	5	11	-12	7	4	5	18	18
-8	7	4	12	-10	-10	1	5	13	-13	-4	2	5	41	-39	0	3	5	10	-9	8	4	5	6	-7
-4	7	4	8	-10	-9	1	5	27	25	-3	2	5	6	-7	1	3	5	13	14	9	4	5	8	9
-3	7	4	7	6	-8	1	5	23	-25	-2	2	5	39	-41	2	3	5	15	15	10	4	5	13	12
-1	7	4	11	12	-7	1	5	18	19	-1	2	5	4	-4	3	3	5	7	8	11	4	5	9	9
0	7	4	12	-10	-5	1	5	27	30	0	2	5	12	-14	5	3	5	13	-12	12	4	5	16	16
1	7	4	7	-6	-4	1	5	20	20	1	2	5	12	-11	6	3	5	4	4	13	4	5	17	18
2	7	4	12	-12	-3	1	5	57	57	3	2	5	53	-53	7	3	5	10	10	14	4	5	5	6
3	7	4	5	-5	-2	1	5	53	50	4	2	5	38	-35	8	3	5	10	-10	15	4	5	7	7
5	7	4	5	-5	-1	1	5	54	54	5	2	5	36	-37	9	3	5	8	-8	-14	5	5	8	8
6	7	4	12	11	0	1	5	10	8	6	2	5	30	28	10	3	5	18	-19	-12	5	5	4	4
8	7	4	5	6	1	1	5	22	27	7	2	5	17	-16	11	3	5	15	-15	-11	5	5	9	9
9	7	4	8	9	3	1	5	51	52	8	2	5	11	-10	12	3	5	22	-24	-7	5	5	8	8
-4	8	4	8	-8	4	1	5	29	30	9	2	5	6	7	13	3	5	16	-16	-6	5	5	17	16
-3	8	4	4	4	5	1	5	7	7	10	2	5	6	-5	15	3	5	5	-5	-5	5	5	8	-9
-2	8	4	7	-5	6	1	5	32	31	11	2	5	19	18	-15	4	5	5	-4	-4	5	5	18	17
0	8	4	6	-6	7	1	5	29	-27	12	2	5	5	-6	-14	4	5	9	9	-3	5	5	11	10
1	8	4	9	-9	8	1	5	27	27	13	2	5	6	5	-13	4	5	11	-11	-2	5	5	16	18
-13	0	5	12	13	10	1	5	7	7	14	2	5	6	6	-12	4	5	5	3	-1	5	5	14	16
-11	0	5	54	53	11	1	5	15	15	15	2	5	14	-14	-11	4	5	7	-6	2	5	5	32	-33
-9	0	5	49	47	12	1	5	7	-7	16	2	5	10	10	-10	4	5	12	12	3	5	5	9	7
-7	0	5	19	-20	13	1	5	15	15	17	2	5	4	-5	-8	4	5	14	-13	4	5	5	32	-34
-5	0	5	22	-21	14	1	5	7	8	-15	3	5	7	-7	-7	4	5	9	-7	5	5	5	8	-8
1	0	5	89	87	15	1	5	7	-7	-14	3	5	12	-13	-6	4	5	21	-20	6	5	5	16	-15
3	0	5	29	29	17	1	5	9	-8	-13	3	5	15	-15	-5	4	5	5	-5	8	5	5	6	6
5	0	5	17	-15	-15	2	5	22	23	-11	3	5	10	-10	-4	4	5	17	-19	9	5	5	8	-8
7	0	5	23	-23	-13	2	5	16	16	-10	3	5	12	12	-3	4	5	17	15	10	5	5	4	-6
9	0	5	16	-17	-12	2	5	9	-8	-8	3	5	23	25	-2	4	5	9	8	12	5	5	4	-6
11	0	5	31	-29	-11	2	5	5	-5	-7	3	5	23	23	1	4	5	30	33	13	5	5	7	7
13	0	5	5	4	-10	2	5	28	-29	-6	3	5	7	-5	2	4	5	14	-13	14	5	5	4	-5
-17	1	5	5	-5	-9	2	5	27	-27	-5	3	5	28	31	3	4	5	25	26	-12	6	5	7	-6
-14	1	5	18	-18	-8	2	5	46	-46	-4	3	5	13	-14	4	4	5	8	7	-9	6	5	10	-8
-13	1	5	28	27	-7	2	5	3	4	-3	3	5	5	5	5	4	5	10	8	-7	6	5	8	-8

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-6	6	5	27	26	-16	0	6	17	17	6	1	6	18	20	10	2	6	19	-18	-5	4	6	10	11
-4	6	5	13	14	-14	0	6	9	9	8	1	6	8	7	13	2	6	10	10	-4	4	6	6	-5
-3	6	5	10	-10	-12	0	6	35	33	9	1	6	6	6	14	2	6	9	9	-3	4	6	9	-9
-2	6	5	6	-6	-10	0	6	26	26	10	1	6	6	-8	16	2	6	6	4	-1	4	6	37	-39
-1	6	5	5	-4	-6	0	6	23	25	11	1	6	13	13	-15	3	6	6	7	1	4	6	26	-27
0	6	5	4	-6	-4	0	6	27	26	12	1	6	22	-21	-10	3	6	4	4	2	4	6	11	11
1	6	5	7	6	-2	0	6	6	7	13	1	6	17	16	-9	3	6	10	-10	5	4	6	14	-13
3	6	5	19	17	0	0	6	25	29	15	1	6	7	6	-8	3	6	5	-6	6	4	6	7	7
5	6	5	12	12	2	0	6	51	46	17	1	6	4	5	-7	3	6	12	12	8	4	6	22	22
6	6	5	28	30	4	0	6	16	-18	-16	2	6	10	-10	-6	3	6	26	25	10	4	6	15	15
7	6	5	12	-12	8	0	6	41	37	-15	2	6	9	7	-5	3	6	31	32	11	4	6	10	-11
9	6	5	8	8	10	0	6	5	-3	-14	2	6	8	-9	-4	3	6	28	30	12	4	6	7	6
10	6	5	6	-7	12	0	6	47	-45	-13	2	6	18	18	-3	3	6	29	30	13	4	6	9	-8
11	6	5	9	9	14	0	6	5	-3	-12	2	6	5	5	-2	3	6	52	55	14	4	6	9	11
-8	7	5	8	8	16	0	6	16	-15	-11	2	6	6	7	-1	3	6	28	-29	-11	5	6	9	7
-7	7	5	6	-8	-15	1	6	7	7	-10	2	6	23	24	0	3	6	29	29	-10	5	6	6	6
-6	7	5	6	-5	-14	1	6	21	-21	-9	2	6	16	-15	2	3	6	37	37	-9	5	6	23	23
-5	7	5	13	-11	-13	1	6	13	-14	-8	2	6	10	9	3	3	6	21	-20	-8	5	6	14	-14
-3	7	5	17	-17	-12	1	6	35	-34	-7	2	6	8	-9	4	3	6	32	32	-7	5	6	21	22
-2	7	5	9	-10	-11	1	6	26	-26	-6	2	6	42	-42	5	3	6	8	-8	-5	5	6	17	18
-1	7	5	10	-11	-9	1	6	21	-21	-5	2	6	26	-27	6	3	6	30	29	-4	5	6	11	-12
0	7	5	9	-10	-8	1	6	5	5	-4	2	6	44	-42	7	3	6	6	5	-3	5	6	6	4
1	7	5	5	-7	-7	1	6	13	-12	-3	2	6	11	-11	8	3	6	5	6	-2	5	6	17	-18
2	7	5	6	6	-6	1	6	13	12	-2	2	6	7	5	9	3	6	10	11	0	5	6	8	-8
3	7	5	15	-15	-4	1	6	22	21	-1	2	6	15	15	10	3	6	21	22	1	5	6	6	7
5	7	5	11	-10	-3	1	6	30	26	0	2	6	23	-22	12	3	6	22	22	2	5	6	14	-13
6	7	5	5	4	-2	1	6	43	-41	1	2	6	7	5	13	3	6	9	-9	3	5	6	18	19
8	7	5	8	-8	-1	1	6	12	-13	3	2	6	25	-29	15	3	6	14	-14	4	5	6	15	-15
9	7	5	6	5	0	1	6	39	-38	4	2	6	30	30	-14	4	6	5	-3	5	5	6	11	-12
-2	8	5	14	15	1	1	6	14	14	5	2	6	37	-37	-13	4	6	7	-7	6	5	6	4	3
-1	8	5	7	7	2	1	6	54	-56	6	2	6	3	2	-11	4	6	7	-7	7	5	6	23	-23
0	8	5	12	12	3	1	6	15	-15	7	2	6	5	-6	-10	4	6	4	-5	8	5	6	9	-10
1	8	5	4	-3	4	1	6	9	-9	8	2	6	32	-33	-8	4	6	14	-14	9	5	6	13	-13
2	8	5	15	16	5	1	6	15	-15	9	2	6	27	-27	-6	4	6	7	-6	10	5	6	5	-4

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
11	5	6	8	-7	-5	0	7	11	-10	16	1	7	5	5	-8	3	7	17	19	1	4	7	6	-6
12	5	6	8	-10	-3	0	7	6	-4	-15	2	7	10	11	-7	3	7	7	7	2	4	7	4	-5
-9	6	6	5	-4	-1	0	7	52	-56	-13	2	7	11	-12	-6	3	7	4	-4	4	4	7	25	-26
-6	6	6	20	-21	1	0	7	81	80	-12	2	7	10	9	-5	3	7	7	-9	5	4	7	4	-2
-5	6	6	6	-7	3	0	7	70	71	-11	2	7	17	-18	-4	3	7	11	-12	6	4	7	16	-15
-4	6	6	16	-18	5	0	7	43	41	-10	2	7	18	17	-3	3	7	5	-5	7	4	7	5	-4
-3	6	6	7	5	7	0	7	21	-22	-9	2	7	6	-5	-2	3	7	19	19	9	4	7	12	-13
-2	6	6	16	-16	9	0	7	36	-34	-8	2	7	8	-7	-1	3	7	12	13	13	4	7	4	5
-1	6	6	31	32	15	0	7	7	-9	-7	2	7	15	15	0	3	7	12	13	14	4	7	4	3
0	6	6	6	-5	17	0	7	13	-11	-6	2	7	12	13	1	3	7	14	-14	-13	5	7	5	6
1	6	6	30	32	-16	1	7	5	4	-5	2	7	11	11	2	3	7	4	3	-12	5	7	5	-4
2	6	6	4	-3	-15	1	7	7	-7	-4	2	7	5	5	3	3	7	9	8	-10	5	7	6	6
3	6	6	13	13	-13	1	7	8	8	-3	2	7	16	17	4	3	7	14	-14	-8	5	7	5	-6
5	6	6	8	-8	-11	1	7	7	-7	-2	2	7	5	-7	5	3	7	27	28	-7	5	7	7	-8
6	6	6	16	15	-10	1	7	10	-10	0	2	7	11	-11	6	3	7	16	16	-6	5	7	6	5
7	6	6	11	11	-9	1	7	13	-14	1	2	7	14	12	7	3	7	32	32	-5	5	7	12	-11
8	6	6	6	7	-8	1	7	30	-31	2	2	7	8	-7	9	3	7	10	11	-4	5	7	38	39
11	6	6	10	10	-7	1	7	16	-15	3	2	7	29	25	11	3	7	7	7	-3	5	7	4	-3
-6	7	6	5	6	-6	1	7	12	-12	4	2	7	19	-20	14	3	7	6	7	-2	5	7	38	41
-5	7	6	12	12	-2	1	7	21	21	5	2	7	9	9	15	3	7	4	3	-1	5	7	8	-9
-3	7	6	8	10	0	1	7	28	-30	6	2	7	18	-20	-14	4	7	5	5	2	5	7	9	-10
-2	7	6	14	-13	1	1	7	44	48	7	2	7	14	15	-12	4	7	10	11	3	5	7	9	10
-1	7	6	6	8	2	1	7	26	-25	8	2	7	29	-28	-11	4	7	6	-5	4	5	7	4	3
0	7	6	15	-14	3	1	7	12	-11	9	2	7	23	23	-10	4	7	15	16	5	5	7	12	-12
1	7	6	6	-8	4	1	7	39	-35	10	2	7	20	-20	-9	4	7	7	-6	7	5	7	9	8
2	7	6	11	-11	5	1	7	43	-44	12	2	7	22	-22	-8	4	7	6	-6	8	5	7	16	16
3	7	6	11	-11	6	1	7	40	-38	13	2	7	12	-12	-7	4	7	5	-8	9	5	7	9	-8
4	7	6	6	-6	7	1	7	16	17	14	2	7	10	-10	-6	4	7	6	4	12	5	7	11	-11
5	7	6	6	-6	8	1	7	4	3	15	2	7	8	-8	-5	4	7	9	-11	-6	6	7	9	10
-17	0	7	10	-12	9	1	7	35	37	-15	3	7	7	-7	-4	4	7	12	11	-5	6	7	11	-11
-13	0	7	10	-7	10	1	7	10	11	-13	3	7	16	-17	-3	4	7	31	-30	-4	6	7	9	-7
-11	0	7	23	22	11	1	7	26	26	-12	3	7	13	13	-2	4	7	19	20	-3	6	7	12	-13
-9	0	7	45	43	13	1	7	21	20	-11	3	7	18	-18	-1	4	7	48	-47	-2	6	7	11	-11
-7	0	7	11	12	14	1	7	17	18	-10	3	7	13	14	0	4	7	24	25	-1	6	7	11	-11

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	6	7	5	-7	-15	1	8	11	11	0	2	8	23	23	-7	4	8	20	22	-8	6	8	7	7
1	6	7	12	-12	-14	1	8	19	-19	1	2	8	4	-5	-6	4	8	7	-5	-7	6	8	15	-16
2	6	7	6	5	-12	1	8	20	-20	2	2	8	35	38	-5	4	8	18	18	-5	6	8	6	-6
4	6	7	15	16	-11	1	8	9	10	3	2	8	18	17	-4	4	8	12	-13	-3	6	8	4	5
5	6	7	7	6	-9	1	8	12	12	4	2	8	23	23	-3	4	8	4	3	-2	6	8	12	-11
6	6	7	17	18	-8	1	8	13	-12	5	2	8	8	-7	-1	4	8	17	-18	-1	6	8	12	14
8	6	7	6	-7	-6	1	8	5	5	6	2	8	16	14	0	4	8	31	-32	0	6	8	11	-12
9	6	7	7	8	-5	1	8	4	-5	7	2	8	4	3	1	4	8	6	-5	1	6	8	4	6
10	6	7	6	-6	-4	1	8	16	-16	8	2	8	7	-7	2	4	8	26	-25	4	6	8	7	-6
-6	7	7	6	-6	-3	1	8	22	-22	9	2	8	18	-18	3	4	8	6	7	5	6	8	22	-21
-5	7	7	11	11	-2	1	8	60	-61	11	2	8	13	-14	4	4	8	14	-15	7	6	8	9	9
0	7	7	11	11	-1	1	8	32	-29	13	2	8	8	-9	6	4	8	16	-17	9	6	8	10	10
1	7	7	5	-5	2	1	8	39	-38	14	2	8	7	8	7	4	8	4	-5	-6	7	8	7	8
2	7	7	8	10	3	1	8	8	-10	15	2	8	5	-6	8	4	8	5	-5	-4	7	8	5	7
3	7	7	11	-11	4	1	8	13	15	-10	3	8	7	-8	9	4	8	17	-18	-2	7	8	7	6
5	7	7	11	-10	5	1	8	43	-41	-8	3	8	13	-14	10	4	8	8	-8	-1	7	8	5	6
7	7	7	4	-3	6	1	8	24	25	-7	3	8	6	7	11	4	8	9	-10	-13	0	9	6	-5
-14	0	8	12	-11	7	1	8	34	-32	-6	3	8	18	-16	12	4	8	9	-10	-11	0	9	14	-14
-12	0	8	20	-20	9	1	8	13	-13	-4	3	8	25	-23	-11	5	8	5	5	-9	0	9	19	-21
-10	0	8	31	-31	10	1	8	4	-5	-3	3	8	12	12	-9	5	8	6	7	-7	0	9	10	-10
-8	0	8	16	-15	13	1	8	7	7	-1	3	8	18	-20	-8	5	8	18	-18	-5	0	9	8	-7
-6	0	8	11	11	14	1	8	18	18	1	3	8	6	-5	-6	5	8	7	6	-3	0	9	16	16
-4	0	8	5	5	15	1	8	8	8	2	3	8	8	7	-5	5	8	9	-10	-1	0	9	20	-21
-2	0	8	80	81	-15	2	8	8	8	4	3	8	8	-7	-2	5	8	15	-15	1	0	9	29	30
0	0	8	49	-49	-13	2	8	8	9	5	3	8	17	18	-1	5	8	22	23	3	0	9	23	23
2	0	8	45	-44	-11	2	8	11	11	7	3	8	8	8	1	5	8	33	35	5	0	9	6	5
4	0	8	6	6	-10	2	8	7	6	8	3	8	15	14	3	5	8	30	30	7	0	9	15	14
6	0	8	50	52	-9	2	8	20	19	9	3	8	9	9	4	5	8	8	7	9	0	9	16	16
8	0	8	55	54	-7	2	8	7	8	10	3	8	22	24	6	5	8	7	7	11	0	9	36	36
10	0	8	12	9	-5	2	8	23	24	11	3	8	10	10	9	5	8	5	7	15	0	9	8	8
12	0	8	31	-31	-4	2	8	20	22	12	3	8	18	19	10	5	8	4	-2	-15	1	9	5	4
14	0	8	6	-6	-3	2	8	27	30	-13	4	8	5	-5	11	5	8	5	4	-13	1	9	13	11
16	0	8	7	-6	-2	2	8	26	25	-9	4	8	16	16	-10	6	8	5	6	-12	1	9	9	8
-16	1	8	7	-8	-1	2	8	14	15	-8	4	8	12	-10	-9	6	8	5	-4	-11	1	9	7	6

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-10	1	9	22	21	-2	2	9	9	-9	7	3	9	4	-3	7	5	9	19	19	-13	1	10	5	4
-9	1	9	12	-11	-1	2	9	19	19	8	3	9	6	7	8	5	9	15	15	-12	1	10	8	9
-8	1	9	15	16	0	2	9	7	9	9	3	9	7	-7	-8	6	9	7	-7	-11	1	10	8	10
-7	1	9	14	-14	1	2	9	8	-9	10	3	9	8	8	-7	6	9	7	8	-10	1	10	5	4
-5	1	9	8	8	2	2	9	35	34	14	3	9	12	13	-4	6	9	8	-9	-9	1	10	23	23
-4	1	9	9	-9	3	2	9	11	-13	-9	4	9	11	11	-3	6	9	8	7	-8	1	10	4	4
-3	1	9	22	23	4	2	9	39	38	-6	4	9	5	6	-2	6	9	11	-11	-7	1	10	9	7
-2	1	9	14	13	5	2	9	4	4	-5	4	9	12	-14	0	6	9	14	-15	-6	1	10	8	8
-1	1	9	16	15	6	2	9	7	8	-4	4	9	24	27	1	6	9	9	-8	-2	1	10	12	-14
0	1	9	40	-39	7	2	9	8	8	-2	4	9	24	26	2	6	9	8	-8	-1	1	10	19	19
1	1	9	9	8	8	2	9	4	-2	-1	4	9	10	9	5	6	9	9	-10	0	1	10	12	-14
2	1	9	6	-6	9	2	9	8	8	0	4	9	16	15	6	6	9	6	-7	1	1	10	16	14
3	1	9	12	-13	10	2	9	14	-14	1	4	9	22	20	7	6	9	9	-8	2	1	10	6	-6
5	1	9	36	-36	11	2	9	9	-9	2	4	9	13	-14	-2	7	9	4	-2	6	1	10	10	-10
6	1	9	21	-22	12	2	9	7	-8	4	4	9	9	-8	-1	7	9	5	5	8	1	10	19	-19
7	1	9	18	19	13	2	9	8	-7	5	4	9	25	-24	0	7	9	11	11	10	1	10	20	-20
8	1	9	16	-15	-12	3	9	5	6	6	4	9	5	4	1	7	9	4	6	12	1	10	6	-7
9	1	9	31	30	-11	3	9	6	-6	7	4	9	24	-25	2	7	9	6	5	13	1	10	17	-17
10	1	9	8	-8	-10	3	9	8	10	9	4	9	10	-11	3	7	9	6	6	14	1	10	7	6
11	1	9	11	12	-8	3	9	6	6	10	4	9	8	8	-14	0	10	5	-7	15	1	10	6	-5
12	1	9	11	-10	-7	3	9	26	-27	13	4	9	6	-6	-12	0	10	18	-17	-14	2	10	4	3
13	1	9	7	7	-6	3	9	5	-6	-10	5	9	12	-14	-10	0	10	22	-22	-12	2	10	8	7
-15	2	9	8	7	-5	3	9	19	-23	-9	5	9	9	8	-8	0	10	4	4	-10	2	10	7	6
-14	2	9	4	-4	-4	3	9	21	-22	-8	5	9	10	-10	-4	0	10	13	-12	-9	2	10	4	3
-13	2	9	9	-10	-3	3	9	35	-36	-5	5	9	6	-5	-2	0	10	7	9	-8	2	10	17	-19
-12	2	9	4	-6	-2	3	9	6	-6	-4	5	9	4	5	0	0	10	52	-54	-7	2	10	9	-9
-11	2	9	16	-17	-1	3	9	41	-40	-3	5	9	6	6	2	0	10	7	-8	-6	2	10	16	-16
-10	2	9	11	11	0	3	9	4	4	-1	5	9	13	12	4	0	10	8	9	-5	2	10	7	-6
-9	2	9	15	-15	1	3	9	29	-30	0	5	9	19	-20	6	0	10	23	23	-4	2	10	15	-14
-8	2	9	17	19	2	3	9	11	10	1	5	9	14	16	8	0	10	7	7	-3	2	10	14	13
-7	2	9	4	-3	3	3	9	13	-15	3	5	9	11	12	10	0	10	11	11	-2	2	10	11	12
-6	2	9	35	35	4	3	9	6	5	4	5	9	19	19	14	0	10	15	14	-1	2	10	23	21
-5	2	9	22	22	5	3	9	13	-12	5	5	9	5	-4	-15	1	10	6	5	0	2	10	15	16
-3	2	9	11	11	6	3	9	12	13	6	5	9	22	23	-14	1	10	4	-4	1	2	10	16	17

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	2	10	12	13	-11	4	10	6	-6	1	6	10	14	-14	7	1	11	8	-9	1	3	11	7	5
3	2	10	23	24	-9	4	10	9	10	2	6	10	4	-2	8	1	11	10	10	2	3	11	5	-6
4	2	10	12	-12	-5	4	10	4	4	3	6	10	11	-10	9	1	11	16	-17	3	3	11	18	17
5	2	10	5	5	-4	4	10	23	22	4	6	10	10	-10	10	1	11	7	-7	4	3	11	16	-14
6	2	10	26	-26	-3	4	10	12	-13	5	6	10	18	-19	12	1	11	11	-12	5	3	11	9	-10
7	2	10	29	29	-2	4	10	36	37	7	6	10	6	7	13	1	11	7	-7	6	3	11	6	-6
8	2	10	8	-9	0	4	10	4	5	-11	0	11	13	11	-13	2	11	7	-6	7	3	11	17	-17
9	2	10	10	11	1	4	10	17	17	-9	0	11	26	27	-11	2	11	5	-6	8	3	11	5	4
11	2	10	9	11	3	4	10	5	5	-7	0	11	13	-11	-9	2	11	6	-7	9	3	11	16	-17
12	2	10	5	6	4	4	10	7	5	-5	0	11	12	-13	-8	2	11	11	-11	10	3	11	10	10
14	2	10	9	9	8	4	10	7	-6	-3	0	11	13	-13	-7	2	11	9	10	11	3	11	12	-12
-13	3	10	11	-11	10	4	10	5	-5	-1	0	11	4	-4	-6	2	11	14	-17	12	3	11	7	7
-12	3	10	5	4	12	4	10	7	-9	1	0	11	6	-7	-5	2	11	4	-4	-11	4	11	4	5
-11	3	10	11	-12	-9	5	10	5	-4	3	0	11	5	-6	-4	2	11	19	-20	-10	4	11	10	-11
-9	3	10	7	-7	-7	5	10	10	-12	5	0	11	13	-12	-3	2	11	27	-27	-9	4	11	11	11
-8	3	10	16	-16	-6	5	10	9	8	7	0	11	39	-36	-2	2	11	19	-18	-8	4	11	10	-9
-7	3	10	14	16	-5	5	10	10	-10	11	0	11	31	29	-1	2	11	20	-20	-7	4	11	5	3
-6	3	10	15	-15	-4	5	10	4	-5	-14	1	11	7	7	0	2	11	7	7	-5	4	11	6	6
-5	3	10	10	10	-3	5	10	12	-12	-13	1	11	11	10	1	2	11	10	-10	-3	4	11	13	15
-4	3	10	4	6	-2	5	10	12	-10	-11	1	11	13	-13	2	2	11	28	26	-2	4	11	11	10
-3	3	10	14	-17	-1	5	10	11	-12	-10	1	11	10	-11	4	2	11	13	13	-1	4	11	34	34
-2	3	10	9	9	1	5	10	8	7	-8	1	11	5	-5	5	2	11	10	-10	1	4	11	24	23
-1	3	10	31	-32	2	5	10	12	11	-7	1	11	7	8	8	2	11	18	19	2	4	11	17	-16
0	3	10	16	-16	3	5	10	6	7	-6	1	11	11	12	9	2	11	14	-13	3	4	11	5	8
1	3	10	33	-32	4	5	10	14	15	-5	1	11	12	11	10	2	11	16	16	8	4	11	12	11
2	3	10	35	-33	6	5	10	4	-1	-4	1	11	18	16	11	2	11	4	-4	9	4	11	5	4
3	3	10	4	4	9	5	10	7	7	-3	1	11	28	29	-9	3	11	15	16	10	4	11	7	8
4	3	10	11	-11	-7	6	10	6	-6	-2	1	11	23	22	-7	3	11	13	13	-9	5	11	7	7
5	3	10	17	15	-5	6	10	6	6	-1	1	11	6	-6	-6	3	11	10	-10	-7	5	11	9	-8
6	3	10	9	-10	-4	6	10	4	4	2	1	11	7	7	-5	3	11	11	11	-6	5	11	4	-4
7	3	10	12	12	-3	6	10	11	13	3	1	11	24	-24	-4	3	11	11	-9	-4	5	11	7	-9
10	3	10	5	-3	-2	6	10	4	5	4	1	11	27	25	-3	3	11	12	-12	-3	5	11	7	6
12	3	10	6	-6	-1	6	10	5	-5	5	1	11	11	-10	-2	3	11	16	16	-2	5	11	19	-19
-12	4	10	6	6	0	6	10	7	7	6	1	11	26	25	0	3	11	10	-13	0	5	11	14	-12

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	5	11	15	-15	5	1	12	18	19	2	3	12	7	9	3	0	13	35	-35	-5	3	13	5	5
3	5	11	4	-2	6	1	12	25	-26	5	3	12	13	-14	5	0	13	23	-23	-4	3	13	7	8
7	5	11	10	9	7	1	12	8	6	10	3	12	9	-8	9	0	13	7	-6	-3	3	13	6	7
-3	6	11	8	7	8	1	12	24	-23	-10	4	12	5	-5	11	0	13	5	2	-2	3	13	12	9
-2	6	11	9	-9	9	1	12	5	4	-7	4	12	10	-10	-12	1	13	6	-6	-1	3	13	22	22
0	6	11	7	-7	11	1	12	7	6	-6	4	12	12	12	-11	1	13	8	-6	0	3	13	10	9
3	6	11	6	3	12	1	12	7	-8	-5	4	12	14	-14	-10	1	13	6	-6	1	3	13	24	24
4	6	11	5	3	-12	2	12	8	8	-4	4	12	6	5	-9	1	13	5	-4	2	3	13	5	-2
-10	0	12	13	13	-11	2	12	7	-8	-1	4	12	6	-6	-8	1	13	7	-8	4	3	13	14	-13
-8	0	12	19	18	-9	2	12	10	-9	0	4	12	17	-17	-6	1	13	14	-16	-7	4	13	6	-5
-6	0	12	9	11	-7	2	12	25	-23	2	4	12	11	11	-5	1	13	18	17	-5	4	13	4	-5
-4	0	12	17	-15	-6	2	12	7	-8	3	4	12	8	7	-4	1	13	12	-13	-3	4	13	10	-11
-2	0	12	6	-6	-5	2	12	5	-5	4	4	12	24	24	-3	1	13	19	23	-2	4	13	4	-5
0	0	12	46	-48	-2	2	12	8	9	5	4	12	5	-1	1	1	13	10	10	-1	4	13	6	-6
2	0	12	25	-25	-1	2	12	18	-18	6	4	12	8	8	2	1	13	21	20	0	4	13	12	-10
4	0	12	22	-22	0	2	12	4	7	-5	5	12	7	-8	3	1	13	10	9	2	4	13	4	-2
6	0	12	29	-30	1	2	12	25	-21	-4	5	12	8	-6	4	1	13	21	20	5	4	13	5	4
8	0	12	4	-2	2	2	12	11	-10	-2	5	12	5	-4	5	1	13	5	-4	7	4	13	6	5
10	0	12	11	-10	3	2	12	17	-16	-1	5	12	15	14	9	1	13	12	11	-1	5	13	4	-5
12	0	12	9	-8	4	2	12	9	-9	1	5	12	5	-5	10	1	13	4	0	0	5	13	5	-5
-13	1	12	4	-2	5	2	12	8	7	2	5	12	7	-8	12	1	13	10	8	1	5	13	5	-5
-11	1	12	8	-8	6	2	12	7	8	3	5	12	16	-14	-9	2	13	12	10	2	5	13	6	-7
-10	1	12	11	9	7	2	12	22	22	4	5	12	6	6	-7	2	13	14	14	-8	0	14	13	12
-9	1	12	10	-11	8	2	12	6	-6	5	5	12	13	-12	-5	2	13	5	8	-6	0	14	14	16
-8	1	12	11	11	9	2	12	11	12	6	5	12	8	-8	-4	2	13	15	-14	-4	0	14	6	9
-7	1	12	10	-11	10	2	12	4	3	7	5	12	12	-12	-3	2	13	12	-10	-2	0	14	7	6
-6	1	12	6	-5	12	2	12	6	5	0	6	12	5	5	-2	2	13	18	-17	6	0	14	6	6
-4	1	12	5	-6	-8	3	12	11	11	1	6	12	6	5	0	2	13	20	-22	8	0	14	6	-7
-2	1	12	5	3	-7	3	12	5	1	-11	0	13	12	12	1	2	13	6	-8	10	0	14	14	-12
-1	1	12	31	29	-6	3	12	22	22	-9	0	13	4	-2	2	2	13	20	-22	-10	1	14	5	3
0	1	12	16	16	-4	3	12	24	24	-5	0	13	20	16	4	2	13	4	6	-8	1	14	5	-3
1	1	12	22	21	-2	3	12	21	22	-3	0	13	10	9	5	2	13	10	10	-7	1	14	4	-5
3	1	12	18	19	-1	3	12	10	-8	-1	0	13	15	18	6	2	13	9	-7	-6	1	14	10	-8
4	1	12	9	8	0	3	12	14	15	1	0	13	24	-23	-9	3	13	9	8	-5	1	14	11	-11

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	1	14	18	-18	-5	3	14	7	8	9	0	15	9	8	-2	3	15	8	7	6	1	16	6	-4
-2	1	14	20	18	-1	3	14	6	8	-2	1	15	8	-8	-1	3	15	4	-5	7	1	16	5	-5
0	1	14	15	16	3	3	14	4	3	-1	1	15	10	-10	1	3	15	6	-5	-6	2	16	6	-7
2	1	14	14	15	4	3	14	8	7	0	1	15	13	-13	5	3	15	9	9	-5	2	16	9	10
4	1	14	9	9	6	3	14	5	5	2	1	15	16	-15	-3	4	15	11	-10	-3	2	16	11	12
8	1	14	8	8	7	3	14	4	-3	3	1	15	6	5	3	4	15	7	-8	-2	2	16	5	-3
-9	2	14	8	9	8	3	14	10	9	4	1	15	9	-10	-6	0	16	8	-5	-1	2	16	9	8
-8	2	14	5	-4	-6	4	14	13	-12	5	1	15	11	12	-4	0	16	6	-5	0	2	16	4	0
-7	2	14	7	8	-4	4	14	15	-15	7	1	15	8	8	-2	0	16	5	3	2	2	16	4	4
-6	2	14	4	3	-2	4	14	11	-11	8	1	15	4	-3	0	0	16	18	17	4	2	16	5	4
-4	2	14	15	12	0	4	14	7	-7	-8	2	15	10	9	2	0	16	18	18	5	2	16	6	4
-3	2	14	9	-10	1	4	14	11	9	-6	2	15	10	9	4	0	16	13	12	-3	3	16	5	-1
-2	2	14	7	7	2	4	14	5	-5	-5	2	15	10	-8	6	0	16	10	9	-2	3	16	10	-10
-1	2	14	13	-12	3	4	14	7	-4	-4	2	15	4	5	-7	1	16	7	5	-1	3	16	7	6
1	2	14	15	-13	-1	5	14	6	7	-3	2	15	9	-9	-6	1	16	9	-8	0	3	16	8	-9
3	2	14	9	-9	-9	0	15	7	-6	3	2	15	5	6	-5	1	16	7	8	2	3	16	6	-5
5	2	14	7	-9	-5	0	15	19	15	4	2	15	5	-5	-4	1	16	7	-10	-3	0	17	7	-7
7	2	14	8	-8	-3	0	15	13	11	6	2	15	12	-12	-3	1	16	5	-3	-1	0	17	8	10
8	2	14	5	6	-1	0	15	24	27	-6	3	15	11	10	1	1	16	14	-13	-3	1	17	13	-12
9	2	14	7	-5	1	0	15	13	14	-5	3	15	9	-8	3	1	16	13	-13	-1	1	17	10	-8
-9	3	14	5	5	3	0	15	11	9	-4	3	15	10	10	5	1	16	7	-6	3	1	17	7	-8
-7	3	14	5	5	5	0	15	11	10	-3	3	15	6	-9										

ASTRO

APPENDIX SEVEN

STRUCTURE (I)
OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	122	-131	8	3	0	9	-8	-8	1	1	25	-26	5	2	1	4	-2	-1	4	1	25	-26
3	0	0	22	22	0	4	0	134	-133	-7	1	1	19	19	6	2	1	21	-21	0	4	1	8	8
4	0	0	7	5	1	4	0	45	46	-6	1	1	8	-7	7	2	1	6	-6	1	4	1	10	10
5	0	0	15	15	3	4	0	50	-47	-5	1	1	9	-9	8	2	1	24	24	2	4	1	10	11
6	0	0	5	-5	4	4	0	19	18	-4	1	1	22	23	9	2	1	28	-29	3	4	1	36	-34
7	0	0	21	-21	5	4	0	14	14	-3	1	1	23	24	10	2	1	5	4	4	4	1	11	-12
9	0	0	32	30	7	4	0	8	8	-2	1	1	24	24	-10	3	1	6	-6	5	4	1	59	56
10	0	0	17	16	9	4	0	7	-7	-1	1	1	78	-86	-9	3	1	8	7	6	4	1	24	-23
2	1	0	138	146	10	4	0	8	-7	1	1	1	23	-22	-8	3	1	11	12	7	4	1	4	3
3	1	0	6	-6	1	5	0	17	17	2	1	1	119	-124	-7	3	1	6	-5	8	4	1	3	0
4	1	0	16	14	2	5	0	27	-27	3	1	1	61	61	-6	3	1	6	-6	9	4	1	4	-2
5	1	0	18	-16	3	5	0	12	11	4	1	1	34	-33	-5	3	1	15	15	-8	5	1	10	12
6	1	0	11	10	4	5	0	3	-1	5	1	1	31	-28	-4	3	1	27	-29	-7	5	1	9	-9
7	1	0	19	-19	6	5	0	22	-21	6	1	1	53	52	-2	3	1	15	-16	-6	5	1	14	14
8	1	0	12	-11	7	5	0	13	13	7	1	1	40	-39	-1	3	1	41	44	-5	5	1	13	14
9	1	0	8	-9	9	5	0	5	6	8	1	1	4	4	0	3	1	94	-96	-4	5	1	6	-6
10	1	0	5	6	0	6	0	11	-13	9	1	1	14	14	1	3	1	33	33	-3	5	1	7	-6
0	2	0	91	-91	1	6	0	22	21	10	1	1	7	-6	2	3	1	42	42	-2	5	1	9	-10
1	2	0	108	-108	3	6	0	41	-41	-11	2	1	5	5	3	3	1	29	26	-1	5	1	33	32
2	2	0	90	84	4	6	0	60	59	-10	2	1	16	17	4	3	1	3	4	0	5	1	18	-17
3	2	0	87	83	5	6	0	13	14	-9	2	1	19	-19	5	3	1	14	-13	1	5	1	18	17
4	2	0	87	-82	6	6	0	15	-14	-8	2	1	4	3	7	3	1	6	7	2	5	1	34	32
5	2	0	39	-35	1	7	0	13	13	-7	2	1	19	20	9	3	1	12	-12	3	5	1	60	-58
6	2	0	9	10	2	7	0	30	-29	-6	2	1	34	-36	10	3	1	7	6	4	5	1	46	45
7	2	0	4	3	5	7	0	9	-10	-5	2	1	7	7	11	3	1	4	-4	5	5	1	17	18
8	2	0	8	-8	6	7	0	14	14	-4	2	1	25	-27	-10	4	1	9	8	6	5	1	37	-35
11	2	0	5	-4	0	8	0	7	-11	-3	2	1	3	1	-9	4	1	5	-4	7	5	1	22	21
1	3	0	8	-11	2	8	0	12	11	-2	2	1	4	3	-8	4	1	7	7	-9	6	1	4	4
2	3	0	51	52	4	8	0	13	-12	-1	2	1	98	104	-7	4	1	4	3	-8	6	1	7	-6
3	3	0	19	-20	5	8	0	9	-8	0	2	1	35	35	-6	4	1	15	-15	-7	6	1	6	-5
4	3	0	29	27	6	8	0	4	1	1	2	1	131	-131	-5	4	1	4	3	-6	6	1	16	16
5	3	0	18	18	1	9	0	4	5	2	2	1	144	141	-4	4	1	9	10	-4	6	1	5	3
6	3	0	14	-13	-11	1	1	5	-4	3	2	1	21	-19	-3	4	1	8	9	-3	6	1	5	-5
7	3	0	3	-2	-10	1	1	7	7	4	2	1	35	-35	-2	4	1	13	-13	-2	6	1	9	11

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	6	1	10	-10	-10	0	2	32	34	10	1	2	7	7	3	3	2	43	40	-2	5	2	4	0
1	6	1	7	6	-9	0	2	7	-6	11	1	2	10	9	4	3	2	40	-40	-1	5	2	14	-17
2	6	1	24	-23	-8	0	2	40	-41	-11	2	2	7	-6	5	3	2	22	22	0	5	2	3	3
3	6	1	7	6	-7	0	2	41	41	-7	2	2	6	7	6	3	2	18	-17	1	5	2	5	6
4	6	1	31	31	-6	0	2	22	23	-6	2	2	25	-27	7	3	2	29	-27	2	5	2	8	-8
5	6	1	17	-17	-5	0	2	33	-32	-5	2	2	30	-31	8	3	2	25	24	3	5	2	4	6
7	6	1	16	16	-4	0	2	39	43	-4	2	2	12	12	9	3	2	17	-16	4	5	2	4	1
8	6	1	8	-7	-3	0	2	69	68	-3	2	2	20	-21	10	3	2	11	11	6	5	2	14	-14
9	6	1	5	4	-2	0	2	26	-26	-2	2	2	41	-44	11	3	2	10	9	7	5	2	7	7
-6	7	1	8	-7	2	0	2	11	12	-1	2	2	71	73	-10	4	2	9	-10	9	5	2	6	-4
-5	7	1	6	-4	3	0	2	97	96	0	2	2	18	19	-8	4	2	23	24	-9	6	2	5	-4
-3	7	1	6	5	4	0	2	25	-25	1	2	2	13	-13	-7	4	2	33	-34	-8	6	2	9	9
0	7	1	4	-3	5	0	2	15	-15	2	2	2	54	-51	-6	4	2	8	-9	-7	6	2	5	-5
1	7	1	7	-9	6	0	2	76	74	3	2	2	7	-6	-5	4	2	27	28	-6	6	2	22	25
2	7	1	11	12	7	0	2	24	-23	4	2	2	35	-31	-4	4	2	18	-19	-5	6	2	26	28
3	7	1	31	-32	8	0	2	5	8	5	2	2	32	-29	-3	4	2	10	10	-4	6	2	41	-45
4	7	1	5	3	9	0	2	47	46	6	2	2	11	10	-2	4	2	18	19	-2	6	2	19	19
6	7	1	7	-6	10	0	2	19	-19	7	2	2	15	-14	-1	4	2	52	-52	-1	6	2	8	-9
7	7	1	5	4	-10	1	2	20	20	8	2	2	4	-4	0	4	2	12	-12	0	6	2	3	2
8	7	1	5	-2	-7	1	2	13	14	9	2	2	5	3	1	4	2	30	30	1	6	2	3	-3
-5	8	1	5	-4	-6	1	2	5	-4	10	2	2	4	-2	2	4	2	9	9	2	6	2	4	3
-4	8	1	4	-5	-4	1	2	34	34	-11	3	2	6	-6	3	4	2	23	-22	4	6	2	24	25
-2	8	1	9	10	-3	1	2	15	14	-10	3	2	11	11	4	4	2	3	-2	5	6	2	22	22
-1	8	1	4	-4	-2	1	2	78	87	-9	3	2	8	-9	5	4	2	25	25	6	6	2	28	-26
0	8	1	8	-8	-1	1	2	15	16	-7	3	2	23	24	6	4	2	31	-31	7	6	2	14	13
1	8	1	14	14	1	1	2	4	2	-6	3	2	30	-33	7	4	2	22	22	8	6	2	5	4
2	8	1	6	-6	2	1	2	55	53	-5	3	2	12	13	9	4	2	25	-24	9	6	2	6	-4
4	8	1	9	9	3	1	2	14	15	-4	3	2	18	19	10	4	2	10	10	-7	7	2	8	-9
5	8	1	24	-24	4	1	2	61	-59	-3	3	2	54	-57	-9	5	2	4	3	-6	7	2	20	21
6	8	1	14	12	5	1	2	15	16	-2	3	2	4	-4	-8	5	2	7	-7	-3	7	2	14	14
0	9	1	4	4	6	1	2	19	18	-1	3	2	12	14	-7	5	2	14	14	-2	7	2	6	-5
1	9	1	5	-4	7	1	2	7	-7	0	3	2	30	29	-6	5	2	10	-10	1	7	2	3	-2
3	9	1	14	13	8	1	2	24	23	1	3	2	6	4	-5	5	2	18	-19	2	7	2	5	5
4	9	1	8	-8	9	1	2	15	-16	2	3	2	12	-12	-4	5	2	11	-12	3	7	2	13	-13

OBSERVED AND CALCULATED STRUCTURE FACTORS '

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	7	2	12	11	8	1	3	9	-8	3	3	3	68	-63	4	5	3	12	-12	4	8	3	11	-11
5	7	2	11	-9	9	1	3	17	16	4	3	3	10	9	7	5	3	11	11	5	8	3	4	4
6	7	2	9	8	10	1	3	6	-6	5	3	3	38	37	8	5	3	5	4	-4	9	3	11	10
7	7	2	9	9	-10	2	3	6	8	6	3	3	11	-10	9	5	3	11	-12	-2	9	3	9	-8
8	7	2	11	-10	-9	2	3	9	-9	7	3	3	4	-5	10	5	3	8	6	-1	9	3	5	3
-6	8	2	5	-6	-8	2	3	17	17	-9	4	3	6	-6	-9	6	3	5	5	0	9	3	4	5
-5	8	2	4	-4	-7	2	3	4	4	-8	4	3	13	-14	-6	6	3	9	9	2	9	3	8	6
-4	8	2	11	11	-6	2	3	11	-11	-7	4	3	12	12	-5	6	3	17	-17	3	9	3	10	-9
-3	8	2	7	8	-5	2	3	12	-12	-6	4	3	7	-9	-3	6	3	38	38	-11	0	4	12	13
-2	8	2	10	-9	-4	2	3	63	66	-4	4	3	10	10	-1	6	3	14	-14	-9	0	4	24	-25
-1	8	2	6	-9	-3	2	3	51	-54	-3	4	3	27	-28	0	6	3	20	19	-8	0	4	7	-5
5	8	2	7	-6	-2	2	3	59	-58	-2	4	3	10	-10	1	6	3	7	-6	-7	0	4	21	22
6	8	2	7	7	-1	2	3	28	27	-1	4	3	55	54	2	6	3	16	15	-6	0	4	37	-36
-3	9	2	4	6	0	2	3	139	-146	0	4	3	23	-23	4	6	3	8	-8	-5	0	4	5	4
-2	9	2	4	-4	1	2	3	94	92	1	4	3	4	-1	5	6	3	6	4	-4	0	4	4	-3
-1	9	2	8	9	2	2	3	3	1	2	4	3	20	-21	6	6	3	6	6	-3	0	4	65	66
0	9	2	4	-6	3	2	3	3	-3	3	4	3	6	-4	7	6	3	7	6	-2	0	4	3	-2
1	9	2	4	-5	4	2	3	12	-12	4	4	3	34	34	8	6	3	4	-3	0	0	4	35	-37
2	9	2	5	-4	5	2	3	14	11	5	4	3	18	-17	-6	7	3	6	-5	3	0	4	7	-6
4	9	2	5	5	6	2	3	8	8	6	4	3	5	-4	-4	7	3	20	-22	4	0	4	40	-42
-10	1	3	7	6	7	2	3	16	-14	7	4	3	11	-10	-3	7	3	6	7	5	0	4	4	2
-8	1	3	15	-15	8	2	3	4	3	8	4	3	10	9	-1	7	3	7	-9	6	0	4	39	-38
-7	1	3	15	16	9	2	3	9	-10	9	4	3	6	-5	0	7	3	6	8	7	0	4	28	27
-5	1	3	31	-32	11	2	3	15	13	-9	5	3	3	1	1	7	3	4	4	8	0	4	50	52
-4	1	3	24	24	-10	3	3	7	-6	-8	5	3	8	8	2	7	3	14	-15	9	0	4	13	-14
-3	1	3	40	-41	-9	3	3	4	-5	-7	5	3	19	-19	3	7	3	7	8	-11	1	4	7	7
-2	1	3	59	-64	-8	3	3	9	10	-5	5	3	36	37	-6	8	3	11	11	-9	1	4	9	10
0	1	3	53	-49	-5	3	3	14	-13	-4	5	3	30	-32	-4	8	3	9	-9	-8	1	4	17	18
1	1	3	37	-34	-4	3	3	48	52	-3	5	3	8	-8	-3	8	3	7	8	-7	1	4	13	-14
2	1	3	72	71	-2	3	3	38	38	-2	5	3	53	55	-2	8	3	14	-16	-5	1	4	10	9
3	1	3	32	29	-1	3	3	99	-100	-1	5	3	27	-28	0	8	3	15	15	-4	1	4	34	33
4	1	3	8	-6	0	3	3	40	39	1	5	3	6	6	1	8	3	17	-17	-3	1	4	6	-6
5	1	3	36	-35	1	3	3	42	39	2	5	3	28	-27	2	8	3	4	-4	-2	1	4	26	-30
6	1	3	3	1	2	3	3	38	-36	3	5	3	48	48	3	8	3	9	8	-1	1	4	100	106

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	1	4	9	9	-4	3	4	8	-8	-7	5	4	9	10	1	7	4	8	-7	8	1	5	46	48
1	1	4	85	77	-3	3	4	15	-15	-5	5	4	5	-5	2	7	4	13	-13	9	1	5	26	-26
2	1	4	85	79	-2	3	4	16	-17	-4	5	4	5	-6	3	7	4	23	22	10	1	5	8	8
3	1	4	30	26	-1	3	4	75	75	-3	5	4	12	12	4	7	4	19	-18	11	1	5	10	8
4	1	4	21	-18	0	3	4	32	29	-1	5	4	29	-29	5	7	4	22	22	-11	2	5	18	17
5	1	4	40	36	1	3	4	12	14	0	5	4	4	4	6	7	4	10	10	-10	2	5	19	-19
6	1	4	7	-8	2	3	4	28	27	1	5	4	48	-47	7	7	4	7	-6	-9.	2	5	7	-6
7	1	4	4	7	3	3	4	50	-51	2	5	4	11	11	-4	8	4	5	3	-8	2	5	18	18
8	1	4	19	-20	4	3	4	7	-3	3	5	4	4	-5	0	8	4	9	8	-7	2	5	27	-28
9	1	4	21	21	5	3	4	24	-23	4	5	4	9	9	1	8	4	6	8	-6	2	5	7	-6
10	1	4	14	14	6	3	4	12	-11	5	5	4	23	-24	3	8	4	11	-10	-5	2	5	5	6
-10	2	4	5	5	7	3	4	18	18	6	5	4	4	5	-1	9	4	7	-7	-4	2	5	18	-19
-9	2	4	4	4	8	3	4	10	-10	7	5	4	6	-7	0	9	4	4	2	-3	2	5	32	31
-8	2	4	3	-3	9	3	4	9	10	10	5	4	5	-5	2	9	4	5	-4	-2	2	5	23	24
-7	2	4	10	-10	-10	4	4	6	-6	-9	6	4	4	4	3	9	4	9	9	-1	2	5	15	15
-6	2	4	17	-16	-9	4	4	9	9	-8	6	4	4	-3	4	9	4	4	-2	0	2	5	12	-13
-4	2	4	51	53	-8	4	4	4	4	-5	6	4	12	13	-10	1	5	4	-4	1	2	5	8	6
-3	2	4	9	11	-7	4	4	5	6	-4	6	4	21	-22	-9	1	5	18	-18	3	2	5	16	-17
-2	2	4	27	-29	-6	4	4	24	26	-3	6	4	3	4	-8	1	5	15	15	4	2	5	22	-19
-1	2	4	12	-13	-5	4	4	4	-6	-2	6	4	14	14	-7	1	5	15	15	5	2	5	18	18
0	2	4	38	39	-4	4	4	24	-26	-1	6	4	7	-9	-6	1	5	30	-31	6	2	5	35	-36
1	2	4	53	-50	-3	4	4	26	-28	1	6	4	23	25	-5	1	5	16	16	7	2	5	33	33
2	2	4	40	35	-2	4	4	33	32	2	6	4	46	-46	-4	1	5	27	27	8	2	5	30	-30
3	2	4	121	-115	-1	4	4	81	-82	3	6	4	48	48	-3	1	5	26	26	10	2	5	20	19
4	2	4	14	-13	0	4	4	4	3	4	6	4	23	22	-2	1	5	39	-40	11	2	5	14	-14
5	2	4	80	74	1	4	4	46	47	5	6	4	46	-45	-1	1	5	12	-13	-11	3	5	4	-2
6	2	4	6	-7	2	4	4	14	-15	6	6	4	19	19	0	1	5	28	28	-10	3	5	9	8
7	2	4	12	-10	3	4	4	29	28	8	6	4	4	-5	1	1	5	34	-31	-9	3	5	14	15
8	2	4	6	-7	4	4	4	18	20	-7	7	4	4	-3	2	1	5	4	-2	-8	3	5	18	-19
10	2	4	4	-4	5	4	4	33	-32	-6	7	4	7	6	3	1	5	31	30	-7	3	5	11	-11
-11	3	4	7	7	6	4	4	13	12	-5	7	4	7	7	4	1	5	69	-65	-6	3	5	22	21
-8	3	4	7	8	8	4	4	13	-14	-3	7	4	10	11	5	1	5	49	48	-5	3	5	8	8
-6	3	4	17	-18	9	4	4	12	11	-1	7	4	14	-13	6	1	5	9	-8	-4	3	5	37	-38
-5	3	4	29	-29	-8	5	4	10	-11	0	7	4	19	20	7	1	5	24	-24	-3	3	5	40	-40

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	3	5	36	37	-7	5	5	7	7	1	7	5	4	5	6	0	6	28	29	1	2	6	23	23
-1	3	5	26	26	-6	5	5	3	3	2	7	5	15	-15	7	0	6	17	-18	2	2	6	30	-29
0	3	5	13	-13	-5	5	5	19	-19	4	7	5	16	16	8	0	6	34	34	3	2	6	27	25
1	3	5	3	2	-3	5	5	23	23	5	7	5	14	-14	9	0	6	28	-29	4	2	6	4	-3
2	3	5	20	19	-2	5	5	11	-12	6	7	5	8	8	10	0	6	10	-9	5	2	6	3	0
4	3	5	8	-7	-1	5	5	17	-17	7	7	5	4	4	11	0	6	16	15	6	2	6	9	-10
5	3	5	3	2	1	5	5	29	29	-5	8	5	7	-7	-11	1	6	11	12	8	2	6	13	12
6	3	5	3	-4	2	5	5	21	-21	-4	8	5	7	6	-8	1	6	14	14	10	2	6	4	-4
8	3	5	15	-16	3	5	5	9	-9	-3	8	5	4	-2	-7	1	6	7	-6	11	2	6	6	4
9	3	5	15	16	4	5	5	42	42	-2	8	5	4	-3	-6	1	6	5	6	-11	3	6	11	11
10	3	5	7	-6	5	5	5	48	-46	2	8	5	9	-9	-5	1	6	37	38	-10	3	6	4	-5
11	3	5	4	-3	6	5	5	21	20	3	8	5	8	8	-4	1	6	14	-14	-9	3	6	14	-14
-10	4	5	5	-4	7	5	5	11	10	4	8	5	17	-17	-3	1	6	7	-6	-8	3	6	15	15
-8	4	5	7	6	8	5	5	22	-21	5	8	5	4	5	-2	1	6	73	75	-7	3	6	25	-25
-7	4	5	15	-15	9	5	5	8	7	6	8	5	8	6	-1	1	6	105	105	-6	3	6	8	-8
-6	4	5	8	7	-7	6	5	17	16	-2	9	5	5	5	0	1	6	78	77	-5	3	6	35	34
-5	4	5	15	14	-5	6	5	4	-1	0	9	5	4	-3	1	1	6	78	-70	-4	3	6	19	-20
-4	4	5	14	-14	-4	6	5	11	11	1	9	5	4	-5	2	1	6	40	-39	-2	3	6	46	44
-3	4	5	15	-15	-3	6	5	6	5	2	9	5	6	6	3	1	6	24	23	-1	3	6	23	-25
-2	4	5	4	6	-2	6	5	8	-9	4	9	5	6	-6	4	1	6	23	24	0	3	6	7	-7
-1	4	5	17	-19	-1	6	5	3	-4	-11	0	6	6	7	5	1	6	32	32	1	3	6	10	-9
0	4	5	11	10	0	6	5	7	8	-9	0	6	40	-41	6	1	6	22	-22	2	3	6	14	14
1	4	5	23	-21	3	6	5	18	18	-8	0	6	27	28	7	1	6	13	13	4	3	6	36	-36
2	4	5	15	15	5	6	5	5	-4	-6	0	6	22	-22	8	1	6	19	20	5	3	6	12	11
3	4	5	17	-17	6	6	5	22	22	-5	0	6	9	10	9	1	6	9	8	6	3	6	24	-23
4	4	5	33	32	7	6	5	15	-13	-4	0	6	9	-9	10	1	6	17	16	7	3	6	14	14
5	4	5	18	-19	8	6	5	8	7	-3	0	6	4	-5	11	1	6	9	-10	8	3	6	3	-2
6	4	5	8	-7	9	6	5	6	5	-2	0	6	64	67	-11	2	6	4	-3	9	3	6	7	-5
7	4	5	16	13	-5	7	5	4	4	-1	0	6	93	-96	-8	2	6	11	12	10	3	6	19	18
8	4	5	9	-10	-4	7	5	5	5	1	0	6	56	56	-7	2	6	9	-8	-9	4	6	18	18
9	4	5	4	0	-3	7	5	4	5	2	0	6	60	-58	-5	2	6	35	38	-8	4	6	19	-20
10	4	5	6	4	-2	7	5	7	-7	3	0	6	9	-7	-3	2	6	13	-13	-7	4	6	16	-16
-10	5	5	7	-6	-1	7	5	6	5	4	0	6	13	-13	-1	2	6	38	36	-6	4	6	17	16
-9	5	5	10	10	0	7	5	6	-6	5	0	6	26	25	0	2	6	30	-28	-5	4	6	13	-12

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	4	6	29	28	1	6	6	8	-9	-7	1	7	4	2	7	2	7	28	-28	9	4	7	9	-8
-2	4	6	30	-30	2	6	6	25	27	-6	1	7	20	-20	8	2	7	4	6	-9	5	7	4	1
-1	4	6	28	26	3	6	6	5	5	-5	1	7	13	13	9	2	7	13	15	-8	5	7	6	-6
0	4	6	43	42	4	6	6	13	-12	-4	1	7	3	2	10	2	7	9	9	-7	5	7	7	-8
1	4	6	21	-20	5	6	6	18	-18	-3	1	7	63	-63	11	2	7	6	-5	-6	5	7	22	22
3	4	6	9	-8	6	6	6	4	3	-2	1	7	104	109	-10	3	7	4	-4	-5	5	7	18	-19
4	4	6	11	10	7	6	6	10	8	-1	1	7	33	-35	-9	3	7	6	7	-4	5	7	5	-4
5	4	6	7	6	8	6	6	9	-9	0	1	7	6	-8	-6	3	7	4	-4	-3	5	7	23	23
8	4	6	26	-25	-8	7	6	9	-9	1	1	7	49	48	-5	3	7	14	13	-2	5	7	18	-19
9	4	6	16	15	-7	7	6	14	14	2	1	7	30	-28	-3	3	7	18	16	-1	5	7	18	-18
-9	5	6	5	-3	-6	7	6	4	6	3	1	7	20	-22	-2	3	7	32	-32	0	5	7	21	22
-8	5	6	9	10	-5	7	6	4	0	4	1	7	25	24	-1	3	7	3	-2	1	5	7	32	-32
-6	5	6	23	-24	-4	7	6	7	6	5	1	7	12	-12	0	3	7	17	17	2	5	7	25	25
-5	5	6	9	-8	-3	7	6	7	-7	6	1	7	7	6	1	3	7	18	-19	3	5	7	9	8
-4	5	6	5	-5	-2	7	6	14	-13	7	1	7	15	15	2	3	7	12	-11	4	5	7	33	-32
-3	5	6	8	8	-1	7	6	16	16	8	1	7	5	-5	3	3	7	4	-2	5	5	7	21	21
-2	5	6	12	-11	0	7	6	4	-4	9	1	7	9	9	4	3	7	9	7	7	5	7	14	-14
-1	5	6	7	-7	3	7	6	8	10	10	1	7	8	-7	5	3	7	16	-15	8	5	7	6	-5
1	5	6	15	15	4	7	6	18	18	11	1	7	5	5	6	3	7	5	-6	9	5	7	5	5
2	5	6	4	-4	6	7	6	5	4	-10	2	7	4	-2	8	3	7	7	6	-8	6	7	5	-3
3	5	6	15	-15	7	7	6	11	-10	-9	2	7	8	6	9	3	7	7	-8	-7	6	7	7	7
4	5	6	13	-12	-5	8	6	8	9	-8	2	7	8	8	-9	4	7	10	-11	-4	6	7	13	13
5	5	6	4	-4	-4	8	6	5	5	-7	2	7	15	-16	-8	4	7	11	11	-2	6	7	6	-6
6	5	6	8	9	-3	8	6	9	-9	-6	2	7	6	6	-6	4	7	8	-7	-1	6	7	24	23
7	5	6	4	4	-2	8	6	4	-5	-5	2	7	22	21	-2	4	7	11	11	0	6	7	10	-11
9	5	6	9	-8	-1	8	6	8	10	-4	2	7	32	-33	-1	4	7	16	-16	1	6	7	5	8
-7	6	6	9	9	2	8	6	10	-10	-3	2	7	44	-45	0	4	7	29	-29	3	6	7	12	-12
-6	6	6	13	13	3	8	6	5	4	-2	2	7	42	43	1	4	7	37	36	4	6	7	13	14
-5	6	6	40	-44	5	8	6	4	4	-1	2	7	41	-42	2	4	7	22	-22	5	6	7	14	13
-4	6	6	5	-6	-3	9	6	3	-1	0	2	7	42	40	3	4	7	38	38	6	6	7	10	-9
-3	6	6	20	20	0	9	6	6	-5	2	2	7	32	-32	4	4	7	32	-31	-6	7	7	6	7
-2	6	6	17	-17	2	9	6	4	3	3	2	7	3	2	5	4	7	24	-22	-5	7	7	8	-8
-1	6	6	12	-13	-9	1	7	8	-8	4	2	7	8	7	6	4	7	6	7	-4	7	7	6	5
0	6	6	10	12	-8	1	7	7	7	6	2	7	19	19	7	4	7	7	8	-2	7	7	6	-6

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	7	7	8	8	-2	0	8	18	18	-8	2	8	9	-10	10	3	8	11	-11	-1	6	8	7	7
0	7	7	10	11	-1	0	8	111	-118	-7	2	8	11	11	-10	4	8	7	8	0	6	8	19	19
1	7	7	13	-14	0	0	8	101	-104	-6	2	8	17	17	-7	4	8	14	13	1	6	8	31	-30
2	7	7	4	4	1	0	8	33	34	-5	2	8	12	12	-6	4	8	6	-7	2	6	8	9	8
3	7	7	18	18	2	0	8	5	-5	-4	2	8	15	-14	-5	4	8	7	-8	3	6	8	17	19
4	7	7	8	-8	3	0	8	6	-6	-3	2	8	4	5	-4	4	8	8	-9	4	6	8	54	-54
5	7	7	8	8	4	0	8	23	24	-2	2	8	28	28	-3	4	8	16	18	5	6	8	15	16
7	7	7	4	3	5	0	8	36	-39	-1	2	8	10	10	-2	4	8	25	-24	6	6	8	12	11
-5	8	7	7	-7	6	0	8	16	-16	1	2	8	20	21	-1	4	8	12	13	7	6	8	17	-17
-4	8	7	9	8	7	0	8	60	64	2	2	8	34	-31	0	4	8	27	23	-7	7	8	4	4
-3	8	7	7	-8	8	0	8	68	-72	3	2	8	29	-26	1	4	8	9	-9	-6	7	8	4	3
-2	8	7	6	-6	9	0	8	7	-6	4	2	8	91	86	2	4	8	31	30	-5	7	8	7	-5
-1	8	7	9	9	10	0	8	13	13	5	2	8	3	-5	3	4	8	5	6	-3	7	8	6	-7
0	8	7	10	-8	11	0	8	6	-6	6	2	8	9	-8	4	4	8	26	-26	-1	7	8	10	10
1	8	7	11	-11	-10	1	8	7	-6	7	2	8	12	11	6	4	8	13	12	1	7	8	14	-15
2	8	7	19	18	-8	1	8	7	-7	10	2	8	4	3	7	4	8	29	-28	2	7	8	30	30
3	8	7	15	-16	-5	1	8	9	9	11	2	8	6	-5	8	4	8	31	32	3	7	8	5	-5
5	8	7	6	5	-4	1	8	5	-6	-9	3	8	8	7	-5	5	8	7	7	4	7	8	3	2
6	8	7	4	-5	-3	1	8	8	7	-8	3	8	5	-5	-4	5	8	7	6	5	7	8	12	11
-3	9	7	5	-4	-2	1	8	78	82	-7	3	8	8	-8	-3	5	8	3	2	6	7	8	16	-14
-1	9	7	5	4	-1	1	8	12	-12	-6	3	8	14	-14	-2	5	8	21	-20	7	7	8	5	5
0	9	7	9	-9	0	1	8	13	13	-5	3	8	7	7	-1	5	8	5	5	-3	8	8	4	1
1	9	7	8	6	1	1	8	43	-37	-4	3	8	3	-2	0	5	8	20	-21	2	8	8	4	-2
2	9	7	5	-4	2	1	8	49	41	-2	3	8	34	35	1	5	8	14	14	4	8	8	5	3
3	9	7	5	-4	3	1	8	9	-8	-1	3	8	3	-3	2	5	8	16	-15	-2	9	8	5	-5
-11	0	8	6	-6	4	1	8	37	37	0	3	8	3	-3	3	5	8	5	-6	0	9	8	6	4
-10	0	8	29	-28	5	1	8	13	-14	1	3	8	4	2	5	5	8	12	12	1	9	8	4	-5
-9	0	8	7	8	6	1	8	28	29	2	3	8	17	-17	6	5	8	12	-11	2	9	8	8	9
-8	0	8	12	12	7	1	8	4	-3	3	3	8	33	-29	7	5	8	4	-4	-10	1	9	9	-8
-7	0	8	30	-29	8	1	8	4	3	4	3	8	4	4	9	5	8	4	-4	-9	1	9	4	4
-6	0	8	12	-12	9	1	8	17	17	5	3	8	25	-26	-6	6	8	11	-11	-8	1	9	15	14
-5	0	8	22	22	10	1	8	12	-13	6	3	8	34	32	-5	6	8	9	-10	-7	1	9	15	-14
-4	0	8	37	36	11	1	8	4	3	8	3	8	6	-6	-4	6	8	7	6	-6	1	9	5	5
-3	0	8	45	-48	-11	2	8	11	10	9	3	8	12	10	-2	6	8	9	-9	-5	1	9	16	15

OBSERVED AND CALCULATED STRUCTURE FACTORS :

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	1	9	3	-3	9	2	9	26	26	4	4	9	3	-1	0	7	9	4	2	-6	1	10	24	24
-3	1	9	10	11	10	2	9	18	-17	6	4	9	6	5	2	7	9	6	6	-5	1	10	12	-13
-2	1	9	6	6	-10	3	9	9	9	7	4	9	9	-9	3	7	9	13	14	-4	1	10	22	-21
-1	1	9	5	-5	-9	3	9	7	-7	9	4	9	12	10	4	7	9	11	-11	-3	1	10	30	31
0	1	9	6	-6	-8	3	9	12	-12	-9	5	9	3	-1	6	7	9	9	9	-2	1	10	27	-27
1	1	9	14	13	-7	3	9	9	8	-8	5	9	6	-4	7	7	9	4	-4	-1	1	10	16	16
2	1	9	18	-16	-5	3	9	8	-8	-7	5	9	8	9	-5	8	9	8	7	0	1	10	4	3
4	1	9	10	9	-4	3	9	22	-21	-6	5	9	19	-19	-2	8	9	8	7	1	1	10	17	-17
5	1	9	3	3	-3	3	9	14	15	-4	5	9	9	9	0	8	9	4	-2	3	1	10	4	3
6	1	9	29	-30	-2	3	9	9	9	-3	5	9	12	-12	2	8	9	5	4	4	1	10	28	-28
7	1	9	51	51	-1	3	9	3	2	-2	5	9	13	-13	4	8	9	4	4	5	1	10	7	-6
8	1	9	20	-19	0	3	9	13	-13	-1	5	9	14	14	-1	9	9	5	-5	6	1	10	6	5
9	1	9	12	-12	1	3	9	13	13	1	5	9	12	-11	-10	0	10	8	-9	7	1	10	15	16
10	1	9	23	23	2	3	9	15	13	2	5	9	11	-11	-8	0	10	21	20	8	1	10	6	7
11	1	9	9	-10	3	3	9	31	-29	3	5	9	20	20	-7	0	10	18	-17	9	1	10	10	8
-11	2	9	5	-4	4	3	9	12	12	4	5	9	20	-20	-6	0	10	5	-3	10	1	10	8	-9
-10	2	9	14	-13	5	3	9	5	3	6	5	9	14	13	-5	0	10	20	-19	-7	2	10	4	-3
-9	2	9	18	17	6	3	9	7	9	7	5	9	28	-27	-4	0	10	28	-29	-6	2	10	31	31
-8	2	9	20	-21	7	3	9	24	-24	8	5	9	8	8	-3	0	10	34	34	-5	2	10	7	7
-7	2	9	6	-5	8	3	9	18	17	9	5	9	7	6	-2	0	10	86	-91	-4	2	10	3	-3
-6	2	9	18	18	10	3	9	5	-4	-8	6	9	7	6	-1	0	10	66	-66	-3	2	10	3	1
-5	2	9	8	-8	-10	4	9	5	-5	-5	6	9	8	9	0	0	10	48	47	-2	2	10	7	-7
-4	2	9	10	10	-9	4	9	11	11	-4	6	9	4	-4	1	0	10	67	-65	-1	2	10	12	-13
-3	2	9	4	-4	-8	4	9	10	-10	-3	6	9	10	-10	2	0	10	93	-93	0	2	10	8	8
-2	2	9	9	10	-7	4	9	5	5	-1	6	9	3	3	3	0	10	49	53	1	2	10	23	23
-1	2	9	15	17	-6	4	9	7	6	1	6	9	5	5	4	0	10	6	7	2	2	10	36	33
0	2	9	22	20	-5	4	9	19	-20	2	6	9	4	3	5	0	10	9	-11	3	2	10	33	28
1	2	9	45	-44	-3	4	9	15	16	4	6	9	7	-6	6	0	10	5	-6	4	2	10	20	-20
2	2	9	47	42	-2	4	9	16	-15	5	6	9	10	10	7	0	10	3	4	6	2	10	12	13
3	2	9	7	6	-1	4	9	7	-7	6	6	9	8	-8	8	0	10	13	-12	7	2	10	5	4
4	2	9	11	-12	0	4	9	5	6	7	6	9	4	4	9	0	10	16	-16	8	2	10	4	-5
5	2	9	25	-24	1	4	9	7	7	8	6	9	8	7	10	0	10	5	5	10	2	10	12	11
6	2	9	20	21	2	4	9	12	-12	-7	7	9	5	5	-10	1	10	8	-8	-10	3	10	11	-10
8	2	9	11	-11	3	4	9	5	-4	-4	7	9	8	7	-7	1	10	4	-4	-9	3	10	7	6

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-8	3	10	6	-5	-6	5	10	6	7	-1	9	10	4	3	6	2	11	6	7	1	5	11	7	8
-7	3	10	18	-18	-2	5	10	4	-3	1	9	10	4	4	7	2	11	12	-11	2	5	11	22	23
-6	3	10	34	33	0	5	10	19	18	-10	1	11	4	3	8	2	11	29	29	3	5	11	39	-39
-5	3	10	5	-5	1	5	10	4	4	-8	1	11	4	-5	10	2	11	7	-5	4	5	11	9	9
-3	3	10	48	49	2	5	10	11	-11	-6	1	11	4	-3	-8	3	11	4	5	5	5	11	16	15
-2	3	10	31	-33	4	5	10	5	4	-5	1	11	18	18	-6	3	11	5	3	6	5	11	27	-27
-1	3	10	11	-9	5	5	10	5	5	-4	1	11	29	-29	-5	3	11	11	-14	8	5	11	10	11
0	3	10	7	-7	8	5	10	5	-5	-3	1	11	27	27	-4	3	11	16	17	-3	6	11	7	-7
1	3	10	32	-31	-8	6	10	4	-3	-2	1	11	8	9	-3	3	11	3	-3	-2	6	11	13	13
3	3	10	3	3	-7	6	10	4	2	-1	1	11	57	-59	-1	3	11	7	7	-1	6	11	13	-13
5	3	10	11	11	-6	6	10	22	-22	0	1	11	72	70	0	3	11	12	-11	1	6	11	10	11
6	3	10	5	6	-4	6	10	23	24	1	1	11	46	-43	1	3	11	9	9	2	6	11	16	-17
7	3	10	5	-6	-3	6	10	17	-18	2	1	11	25	-23	4	3	11	21	-19	3	6	11	8	8
8	3	10	7	-6	-2	6	10	11	-12	3	1	11	47	45	5	3	11	7	6	4	6	11	10	10
9	3	10	9	9	-1	6	10	10	10	4	1	11	11	9	6	3	11	11	-12	5	6	11	19	-18
10	3	10	4	-3	0	6	10	15	-14	5	1	11	20	-21	7	3	11	17	17	6	6	11	9	-8
-10	4	10	6	5	1	6	10	12	12	6	1	11	40	41	-7	4	11	5	-5	7	6	11	10	9
-8	4	10	10	-9	3	6	10	27	-28	7	1	11	20	-20	-3	4	11	7	7	-4	7	11	8	7
-7	4	10	9	9	5	6	10	5	3	8	1	11	8	-9	-1	4	11	14	-15	-3	7	11	7	-6
-5	4	10	10	-10	6	6	10	5	-4	9	1	11	6	6	0	4	11	32	32	-1	7	11	8	9
-4	4	10	18	17	-6	7	10	13	-12	10	1	11	7	-7	1	4	11	23	-22	2	7	11	16	17
-3	4	10	10	-12	-4	7	10	3	-4	-10	2	11	6	6	2	4	11	11	10	3	7	11	9	-9
-2	4	10	33	34	-3	7	10	5	-5	-7	2	11	7	-7	3	4	11	5	-5	4	7	11	7	7
-1	4	10	42	44	-2	7	10	13	12	-6	2	11	8	7	4	4	11	19	-18	5	7	11	5	5
0	4	10	32	-34	-1	7	10	4	-5	-5	2	11	4	4	5	4	11	9	9	-5	8	11	5	5
1	4	10	9	7	0	7	10	12	-11	-4	2	11	30	-29	6	4	11	3	4	-3	8	11	6	-5
3	4	10	17	-18	1	7	10	14	15	-3	2	11	40	40	7	4	11	6	-6	-2	8	11	7	6
4	4	10	8	9	3	7	10	6	8	-2	2	11	29	-31	-7	5	11	4	1	0	8	11	12	-12
5	4	10	10	9	6	7	10	6	-7	-1	2	11	23	24	-5	5	11	11	-10	1	8	11	18	18
6	4	10	12	-11	-4	8	10	6	-6	1	2	11	31	-32	-4	5	11	9	9	2	8	11	8	-8
7	4	10	8	-7	-2	8	10	10	12	2	2	11	4	5	-3	5	11	16	-17	4	8	11	10	9
8	4	10	13	13	1	8	10	7	-7	3	2	11	9	11	-2	5	11	6	-6	-10	0	12	4	3
9	4	10	7	5	3	8	10	5	4	4	2	11	32	-32	-1	5	11	27	28	-9	0	12	14	12
-7	5	10	10	-10	5	8	10	4	-1	5	2	11	37	37	0	5	11	33	-33	-8	0	12	30	-29

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-7	0	12	11	11	-10	2	12	5	-4	-8	4	12	8	6	-6	6	12	4	-4	6	1	13	42	44
-6	0	12	6	6	-9	2	12	6	6	-7	4	12	7	-7	-5	6	12	14	13	7	1	13	7	-8
-5	0	12	16	17	-7	2	12	15	15	-6	4	12	4	4	-4	6	12	7	-7	8	1	13	25	-25
-4	0	12	12	-11	-6	2	12	8	-9	-5	4	12	7	8	-2	6	12	4	4	9	1	13	21	21
-3	0	12	71	-69	-5	2	12	21	-21	-4	4	12	5	-5	-1	6	12	10	9	10	1	13	5	-4
-2	0	12	32	-32	-4	2	12	11	10	-3	4	12	8	7	0	6	12	10	-9	-8	2	13	7	-6
-1	0	12	21	-22	-3	2	12	21	23	-2	4	12	4	3	1	6	12	9	-9	-7	2	13	21	19
0	0	12	89	87	-2	2	12	13	14	-1	4	12	8	10	2	6	12	15	14	-6	2	13	15	-15
1	0	12	8	-8	-1	2	12	14	-14	0	4	12	20	-20	3	6	12	39	-41	-5	2	13	3	3
2	0	12	23	-24	3	2	12	57	52	1	4	12	10	9	4	6	12	10	11	-4	2	13	12	12
3	0	12	13	14	4	2	12	8	-8	2	4	12	3	3	5	6	12	14	14	-3	2	13	14	-14
4	0	12	13	-11	5	2	12	24	-20	3	4	12	24	-24	6	6	12	13	-14	-2	2	13	35	35
5	0	12	41	-43	6	2	12	16	15	4	4	12	5	5	-5	7	12	8	-8	-1	2	13	5	6
6	0	12	59	61	7	2	12	14	14	5	4	12	26	26	-4	7	12	4	-6	0	2	13	32	-30
7	0	12	75	-80	9	2	12	6	6	6	4	12	30	-29	-2	7	12	4	6	1	2	13	20	19
8	0	12	35	-35	10	2	12	10	-10	7	4	12	22	24	0	7	12	11	-12	2	2	13	28	25
9	0	12	38	40	-10	3	12	7	5	8	4	12	11	11	1	7	12	23	24	4	2	13	27	-25
-9	1	12	6	-5	-9	3	12	4	-5	9	4	12	15	-16	3	7	12	15	-16	5	2	13	8	7
-8	1	12	5	-5	-7	3	12	11	12	-8	5	12	4	-4	4	7	12	5	4	6	2	13	11	11
-6	1	12	13	-11	-6	3	12	5	-6	-7	5	12	4	3	5	7	12	12	-13	7	2	13	18	-17
-5	1	12	3	3	-5	3	12	14	13	-6	5	12	8	6	-1	8	12	4	-1	8	2	13	20	20
-4	1	12	17	-17	-4	3	12	4	-6	-4	5	12	7	8	3	8	12	6	4	9	2	13	9	-10
-3	1	12	32	32	-2	3	12	11	-11	-3	5	12	8	-8	-9	1	13	6	4	10	2	13	6	-5
-2	1	12	19	-19	-1	3	12	12	-12	-2	5	12	10	9	-8	1	13	4	-4	-9	3	13	5	-4
-1	1	12	49	-50	0	3	12	16	16	-1	5	12	4	-3	-6	1	13	12	13	-8	3	13	5	4
0	1	12	50	-48	1	3	12	28	-29	0	5	12	19	21	-5	1	13	12	-11	-6	3	13	4	-5
1	1	12	15	14	3	3	12	27	27	1	5	12	10	-12	-4	1	13	12	13	-3	3	13	20	19
2	1	12	37	34	4	3	12	13	-13	2	5	12	12	-13	-2	1	13	18	-20	-2	3	13	8	-8
3	1	12	3	-2	5	3	12	17	15	3	5	12	10	9	-1	1	13	30	31	-1	3	13	18	-19
4	1	12	38	-37	6	3	12	10	10	4	5	12	18	19	0	1	13	14	-14	0	3	13	4	4
5	1	12	26	27	7	3	12	24	-24	5	5	12	10	-11	1	1	13	17	-17	1	3	13	27	30
6	1	12	27	28	8	3	12	16	14	6	5	12	7	-7	3	1	13	19	20	2	3	13	19	-19
7	1	12	29	-31	9	3	12	4	-5	7	5	12	9	9	4	1	13	9	-9	3	3	13	7	4
9	1	12	20	-20	-9	4	12	5	-5	-7	6	12	10	-11	5	1	13	30	-31	4	3	13	18	17

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
5	3	13	20	21	-7	6	13	5	-3	1	0	14	49	-48	7	2	14	13	-13	-2	5	14	5	-6
6	3	13	27	-28	-6	6	13	4	2	2	0	14	48	45	9	2	14	10	10	-1	5	14	14	13
8	3	13	7	8	-5	6	13	4	-5	3	0	14	11	9	-7	3	14	11	10	0	5	14	5	-3
9	3	13	4	-3	-4	6	13	8	-7	4	0	14	32	-33	-6	3	14	4	6	2	5	14	18	17
-8	4	13	6	-7	-3	6	13	7	7	6	0	14	7	7	-5	3	14	16	-15	3	5	14	13	13
-7	4	13	11	11	-2	6	13	6	-6	7	0	14	10	9	-4	3	14	31	30	4	5	14	4	-3
-6	4	13	13	-13	-1	6	13	6	-6	8	0	14	5	-4	-3	3	14	23	-25	5	5	14	6	-5
-4	4	13	20	20	0	6	13	5	5	10	0	14	8	-9	-2	3	14	13	-13	-6	6	14	6	7
-3	4	13	17	-18	1	6	13	4	-4	-10	1	14	4	-2	-1	3	14	31	33	-5	6	14	13	13
-2	4	13	3	0	2	6	13	4	-3	-5	1	14	10	-11	0	3	14	41	-41	-4	6	14	6	-8
-1	4	13	10	9	3	6	13	8	-7	-4	1	14	21	23	1	3	14	11	9	-3	6	14	12	-11
1	4	13	5	-4	4	6	13	6	5	-3	1	14	39	-39	2	3	14	10	9	-2	6	14	17	18
2	4	13	16	-17	5	6	13	5	-5	-2	1	14	23	-23	3	3	14	10	-9	-1	6	14	10	-11
4	4	13	10	9	7	6	13	5	5	0	1	14	25	-26	4	3	14	13	12	0	6	14	7	-7
5	4	13	7	8	-6	7	13	5	-4	1	1	14	5	6	5	3	14	19	19	2	6	14	25	-24
6	4	13	6	-5	-5	7	13	5	4	2	1	14	4	7	6	3	14	8	-8	3	6	14	20	20
8	4	13	5	4	-2	7	13	4	4	3	1	14	31	-31	7	3	14	7	-6	4	6	14	12	12
9	4	13	4	4	1	7	13	4	3	4	1	14	15	-17	8	3	14	4	5	5	6	14	11	-10
-8	5	13	6	4	2	7	13	4	5	6	1	14	5	-4	-6	4	14	4	-5	-5	7	14	5	5
-7	5	13	6	-6	3	7	13	9	-9	7	1	14	5	-7	-5	4	14	6	5	-4	7	14	4	-3
-6	5	13	5	-6	5	7	13	4	4	8	1	14	4	4	-3	4	14	4	3	-3	7	14	10	9
-5	5	13	20	20	-4	8	13	8	-8	9	1	14	4	-2	-2	4	14	26	26	-2	7	14	4	-3
-4	5	13	18	-19	-3	8	13	11	10	-6	2	14	3	-1	-1	4	14	38	-38	-1	7	14	14	-14
-3	5	13	10	-10	2	8	13	5	4	-5	2	14	5	-7	0	4	14	9	9	0	7	14	20	20
-2	5	13	20	21	3	8	13	4	4	-4	2	14	8	8	1	4	14	27	27	1	7	14	8	-7
-1	5	13	6	-5	-10	0	14	8	8	-2	2	14	15	-15	2	4	14	25	-26	2	7	14	11	-11
1	5	13	5	-6	-8	0	14	6	5	-1	2	14	10	10	3	4	14	8	-7	4	7	14	5	-5
2	5	13	6	7	-7	0	14	3	-1	0	2	14	8	8	4	4	14	4	2	-3	8	14	6	6
3	5	13	8	-8	-6	0	14	3	2	1	2	14	6	6	5	4	14	8	-8	-2	8	14	7	-6
4	5	13	6	-6	-5	0	14	21	-20	2	2	14	17	15	7	4	14	10	11	-10	1	15	5	3
5	5	13	7	7	-3	0	14	20	-19	3	2	14	15	-13	-7	5	14	10	10	-9	1	15	7	-6
6	5	13	16	-15	-2	0	14	26	-25	4	2	14	9	6	-6	5	14	5	5	-7	1	15	6	6
7	5	13	9	9	-1	0	14	72	71	5	2	14	15	14	-5	5	14	8	-9	-6	1	15	9	-10
8	5	13	13	11	0	0	14	34	-33	6	2	14	11	-10	-3	5	14	4	6	-4	1	15	4	4

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	1	15	32	-32	-1	3	15	6	-5	-3	6	15	6	6	7	0	16	33	-37	9	2	16	8	-9
-1	1	15	18	17	1	3	15	11	-10	-2	6	15	6	-6	8	0	16	40	43	-9	3	16	4	-3
0	1	15	6	7	2	3	15	4	4	-1	6	15	9	-8	9	0	16	4	5	-8	3	16	15	12
1	1	15	28	-29	3	3	15	15	-16	0	6	15	14	15	-8	1	16	4	2	-6	3	16	5	-4
2	1	15	47	43	4	3	15	23	12	1	6	15	8	-9	-7	1	16	15	-13	-5	3	16	13	13
3	1	15	11	10	5	3	15	12	-13	2	6	15	4	-4	-6	1	16	6	-7	-4	3	16	24	-23
4	1	15	32	-31	6	3	15	15	14	3	6	15	7	7	-5	1	16	10	10	-3	3	16	10	-9
5	1	15	35	34	7	3	15	9	9	4	6	15	14	-14	-4	1	16	8	-10	-2	3	16	8	6
6	1	15	19	-19	8	3	15	3	-2	5	6	15	8	-8	-3	1	16	14	-13	-1	3	16	5	-5
7	1	15	24	-24	-9	4	15	5	-4	6	6	15	10	9	-2	1	16	17	-17	0	3	16	4	3
8	1	15	14	13	-6	4	15	7	5	-3	7	15	5	-5	-1	1	16	20	-19	1	3	16	6	6
9	1	15	6	-6	-4	4	15	10	-9	-2	7	15	4	3	0	1	16	18	19	2	3	16	40	39
-8	2	15	11	11	-3	4	15	3	2	-1	7	15	5	4	2	1	16	8	-8	3	3	16	12	-11
-7	2	15	11	-11	-1	4	15	33	34	1	7	15	7	8	3	1	16	21	-22	4	3	16	13	-11
-6	2	15	4	4	0	4	15	19	-19	2	7	15	9	-10	4	1	16	12	11	5	3	16	14	13
-4	2	15	19	18	1	4	15	19	-20	3	7	15	4	5	5	1	16	32	34	6	3	16	22	-22
-3	2	15	4	5	2	4	15	20	20	4	7	15	6	6	6	1	16	40	-41	7	3	16	9	7
-2	2	15	24	-24	4	4	15	6	5	-2	8	15	6	3	7	1	16	10	-10	-6	4	16	15	14
-1	2	15	4	-5	6	4	15	9	-7	-1	8	15	9	-10	8	1	16	8	-8	-5	4	16	4	-5
0	2	15	17	-16	7	4	15	5	4	0	8	15	12	11	9	1	16	4	-3	-4	4	16	3	-5
1	2	15	11	11	8	4	15	4	4	-9	0	16	8	-8	-9	2	16	5	3	-3	4	16	9	10
2	2	15	22	20	-7	5	15	4	-4	-7	0	16	19	17	-8	2	16	4	-2	-2	4	16	12	-14
3	2	15	42	-41	-5	5	15	4	-5	-6	0	16	20	-19	-6	2	16	15	-14	-1	4	16	14	-14
4	2	15	22	22	-2	5	15	15	15	-5	0	16	8	7	-5	2	16	4	4	0	4	16	8	-7
5	2	15	39	39	-1	5	15	18	-18	-3	0	16	5	-4	-4	2	16	7	5	1	4	16	7	-7
6	2	15	17	-19	0	5	15	5	-6	-2	0	16	37	40	-3	2	16	12	-13	4	4	16	30	31
7	2	15	12	13	1	5	15	33	34	-1	0	16	14	15	-1	2	16	6	7	5	4	16	22	-21
8	2	15	7	-7	2	5	15	33	-33	0	0	16	8	-11	0	2	16	15	16	6	4	16	4	3
9	2	15	4	-6	3	5	15	6	-5	1	0	16	14	15	1	2	16	12	-12	7	4	16	13	13
-9	3	15	4	4	4	5	15	25	24	2	0	16	15	16	3	2	16	12	-11	8	4	16	17	-18
-8	3	15	5	-6	5	5	15	20	-20	3	0	16	12	13	4	2	16	14	-13	-7	5	16	6	5
-6	3	15	6	7	7	5	15	16	17	4	0	16	50	-53	5	2	16	11	9	-6	5	16	7	-5
-5	3	15	3	2	-6	6	15	6	-6	5	0	16	34	37	6	2	16	11	10	-4	5	16	3	4
-2	3	15	19	20	-5	6	15	5	-6	6	0	16	24	-24	8	2	16	8	7	-3	5	16	6	7

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	5	16	8	6	-8	2	17	4	3	1	4	17	7	6	4	0	18	28	26	-1	3	18	15	-15
1	5	16	16	-17	-7	2	17	4	-4	2	4	17	6	-5	5	0	18	9	-8	0	3	18	9	-10
3	5	16	10	9	-6	2	17	5	-3	3	4	17	8	9	6	0	18	5	9	1	3	18	30	32
4	5	16	4	-2	-5	2	17	22	22	6	4	17	6	8	7	0	18	19	19	2	3	18	25	-26
5	5	16	9	-8	-4	2	17	22	-23	-7	5	17	4	-3	8	0	18	5	-5	3	3	18	4	2
6	5	16	12	13	-3	2	17	8	-6	-6	5	17	8	7	-6	1	18	7	6	4	3	18	18	17
-6	6	16	10	8	-2	2	17	37	38	-4	5	17	12	-11	-5	1	18	12	12	5	3	18	15	-13
-4	6	16	5	-4	-1	2	17	24	-27	-3	5	17	25	24	-4	1	18	20	-20	6	3	18	7	6
-3	6	16	12	13	0	2	17	7	6	-2	5	17	10	-9	-3	1	18	21	-23	7	3	18	5	5
-2	6	16	4	-4	1	2	17	12	14	-1	5	17	13	-14	-2	1	18	26	23	-4	4	18	4	-3
0	6	16	4	-3	2	2	17	15	-15	0	5	17	14	14	0	1	18	8	-9	-3	4	18	6	6
1	6	16	11	12	3	2	17	8	6	1	5	17	5	-5	2	1	18	28	-27	-2	4	18	15	-15
2	6	16	9	-9	4	2	17	8	-9	2	5	17	5	3	3	1	18	7	9	-1	4	18	5	-6
4	6	16	12	11	5	2	17	7	9	6	5	17	7	6	4	1	18	14	14	0	4	18	32	32
5	6	16	7	-7	6	2	17	14	-16	-5	6	17	5	-3	5	1	18	18	-19	1	4	18	14	-12
-4	7	16	8	7	-7	3	17	4	-3	-4	6	17	4	4	6	1	18	7	-8	2	4	18	15	-16
-2	7	16	5	-7	-6	3	17	6	6	-2	6	17	10	-10	8	1	18	6	6	3	4	18	10	8
0	7	16	9	10	-4	3	17	5	-6	-1	6	17	8	8	-9	2	18	4	-3	4	4	18	14	-13
2	7	16	17	-17	-2	3	17	12	-11	0	6	17	9	-7	-5	2	18	4	-3	5	4	18	6	6
-7	1	17	4	4	-1	3	17	9	11	1	6	17	5	-5	-3	2	18	4	4	6	4	18	4	3
-6	1	17	9	-7	0	3	17	19	19	4	6	17	4	2	-1	2	18	4	-4	7	4	18	9	-10
-4	1	17	16	15	1	3	17	13	-13	5	6	17	4	-2	0	2	18	16	-16	-6	5	18	6	-7
-3	1	17	24	-23	3	3	17	17	17	1	7	17	6	-6	1	2	18	8	7	-4	5	18	9	8
-2	1	17	12	12	4	3	17	16	15	2	7	17	5	-6	2	2	18	4	-5	1	5	18	21	23
-1	1	17	7	7	5	3	17	14	-14	-8	0	18	7	7	3	2	18	11	-8	2	5	18	5	5
0	1	17	34	-35	6	3	17	11	-10	-5	0	18	8	7	4	2	18	8	9	3	5	18	8	-9
1	1	17	27	28	7	3	17	10	10	-4	0	18	9	7	5	2	18	5	-5	4	5	18	6	-7
3	1	17	8	-8	-8	4	17	9	8	-3	0	18	18	-18	6	2	18	9	-10	-4	6	18	3	-4
4	1	17	17	-17	-6	4	17	9	-9	-2	0	18	27	29	-7	3	18	4	-3	-3	6	18	4	2
5	1	17	13	14	-5	4	17	11	10	-1	0	18	25	25	-6	3	18	8	-7	-1	6	18	11	-9
6	1	17	6	6	-3	4	17	6	-5	0	0	18	29	-30	-5	3	18	7	5	0	6	18	6	6
7	1	17	20	-21	-2	4	17	9	8	1	0	18	19	20	-4	3	18	4	-3	1	6	18	11	-11
8	1	17	11	13	-1	4	17	15	-16	2	0	18	12	14	-3	3	18	14	-13	2	6	18	12	14
-9	2	17	4	-2	0	4	17	4	4	3	0	18	25	-24	-2	3	18	34	34	3	6	18	21	22

OBSERVED AND CALCULATED STRUCTURE FACTORS .

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	6	18	14	-13	-5	3	19	6	-5	-8	0	20	7	6	3	2	20	4	-4	-1	1	21	17	-17
-2	7	18	8	-8	-4	3	19	5	6	-7	0	20	5	-5	6	2	20	4	-4	1	1	21	10	12
-1	7	18	14	15	-3	3	19	15	15	-6	0	20	9	-8	7	2	20	5	3	2	1	21	16	-16
0	7	18	4	-3	-2	3	19	6	-7	-5	0	20	17	16	-7	3	20	9	-7	3	1	21	6	4
1	7	18	20	-21	-1	3	19	15	-15	-4	0	20	11	10	-6	3	20	6	4	5	1	21	5	-2
2	7	18	7	5	1	3	19	13	12	-3	0	20	19	18	-5	3	20	4	-1	7	1	21	5	5
-8	1	19	4	3	2	3	19	4	-3	-2	0	20	18	19	-4	3	20	10	-10	-7	2	21	4	-2
-7	1	19	5	-5	4	3	19	12	-13	-1	0	20	25	-24	-3	3	20	10	12	-6	2	21	8	7
-5	1	19	8	8	5	3	19	8	7	0	0	20	8	8	-2	3	20	7	-8	-5	2	21	6	-6
-4	1	19	10	-11	6	3	19	14	16	1	0	20	7	6	0	3	20	18	18	-4	2	21	14	-12
-3	1	19	17	-15	7	3	19	7	-6	3	0	20	13	-13	1	3	20	4	6	-3	2	21	22	21
-2	1	19	13	13	-6	4	19	5	3	4	0	20	11	11	3	3	20	13	-14	-1	2	21	16	-16
-1	1	19	9	10	-5	4	19	4	-3	5	0	20	13	13	4	3	20	8	7	0	2	21	17	16
0	1	19	4	-4	-4	4	19	4	4	6	0	20	17	-18	5	3	20	8	-8	1	2	21	9	-10
1	1	19	3	4	-3	4	19	4	3	7	0	20	23	26	6	3	20	6	-4	4	2	21	13	-14
2	1	19	13	13	-2	4	19	6	4	-5	1	20	15	-14	7	3	20	7	7	6	2	21	5	-4
3	1	19	28	-27	-1	4	19	7	-5	-4	1	20	15	-14	-5	4	20	6	-6	7	2	21	4	-2
4	1	19	23	24	0	4	19	15	-15	-3	1	20	25	25	-4	4	20	4	3	-3	3	21	6	-5
5	1	19	7	-8	1	4	19	22	21	-2	1	20	13	12	-2	4	20	15	-14	-2	3	21	6	-8
6	1	19	29	-30	2	4	19	7	6	0	1	20	3	4	0	4	20	7	-8	-1	3	21	14	15
7	1	19	23	22	3	4	19	11	-9	1	1	20	7	-7	1	4	20	5	5	-6	4	21	5	2
8	1	19	5	-4	5	4	19	5	-5	2	1	20	9	7	3	4	20	12	11	-5	4	21	4	3
-7	2	19	4	-3	-6	5	19	4	3	3	1	20	4	-3	5	4	20	7	-7	-4	4	21	6	-5
-6	2	19	7	7	-4	5	19	5	5	4	1	20	9	11	6	4	20	8	9	0	4	21	12	14
-4	2	19	6	-8	0	5	19	14	15	5	1	20	19	-21	-5	5	20	5	6	1	4	21	7	-7
-3	2	19	10	-9	1	5	19	16	-16	7	1	20	7	8	1	5	20	6	-7	3	4	21	11	10
0	2	19	5	-5	2	5	19	11	-13	-5	2	20	5	2	2	5	20	5	-4	4	4	21	7	-6
1	2	19	12	12	3	5	19	27	26	-4	2	20	12	-11	5	5	20	9	9	5	4	21	4	5
2	2	19	12	-11	4	5	19	5	-4	-3	2	20	7	-6	-4	6	20	4	5	-5	5	21	6	-4
3	2	19	4	-3	5	5	19	6	-3	-2	2	20	5	4	-1	6	20	5	6	-4	5	21	5	3
4	2	19	42	40	-1	6	19	10	10	-1	2	20	7	6	0	6	20	5	4	-2	5	21	9	-8
5	2	19	26	-25	1	6	19	11	-9	0	2	20	4	-3	-5	1	21	7	3	-1	5	21	12	11
7	2	19	4	-4	4	6	19	7	-6	1	2	20	6	-6	-4	1	21	5	-4	0	5	21	6	-6
-7	3	19	7	7	1	7	19	5	-5	2	2	20	7	-7	-2	1	21	15	15	1	5	21	5	-3

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	5	21	14	14	-1	2	22	4	-5	-5	2	23	5	-4	-4	1	24	13	14	4	1	25	7	-7
3	5	21	5	-6	0	2	22	5	4	0	2	23	4	4	-3	1	24	4	7	-4	2	25	6	7
1	6	21	7	7	1	2	22	4	-3	1	2	23	5	6	-2	1	24	6	-5	-2	2	25	3	-3
-6	0	22	4	4	2	2	22	12	-10	2	2	23	11	-11	-1	1	24	7	4	-1	2	25	7	5
-4	0	22	8	-7	3	2	22	4	2	3	2	23	13	14	0	1	24	4	4	0	2	25	5	-4
-3	0	22	6	6	4	2	22	9	6	4	2	23	12	-10	1	1	24	5	8	1	2	25	4	-4
-2	0	22	14	11	5	2	22	6	-5	5	2	23	6	-7	2	1	24	4	-2	2	2	25	8	5
-1	0	22	12	-10	-3	3	22	13	12	-2	3	23	5	-6	5	1	24	4	6	3	2	25	9	-10
0	0	22	10	-10	-1	3	22	9	-9	0	3	23	6	7	-5	2	24	5	-5	-2	3	25	4	6
1	0	22	18	19	0	3	22	15	13	1	3	23	4	-3	-1	2	24	4	-2	-1	3	25	4	3
3	0	22	12	14	1	3	22	11	-11	5	3	23	8	8	2	2	24	6	8	0	3	25	5	-5
5	0	22	15	-14	2	3	22	16	-15	0	4	23	6	3	3	2	24	5	5	1	3	25	4	-3
6	0	22	27	26	3	3	22	19	19	2	4	23	5	-4	4	2	24	7	-7	3	3	25	5	4
-6	1	22	8	7	4	3	22	12	-14	0	5	23	5	-6	-4	3	24	7	6	-1	4	25	7	8
-4	1	22	4	-3	-1	4	22	10	12	2	5	23	9	10	-1	3	24	6	7	-4	0	26	7	-4
-3	1	22	11	11	1	4	22	11	-12	-6	0	24	5	6	0	3	24	8	-8	-3	0	26	7	-5
-1	1	22	9	-12	2	4	22	8	8	-5	0	24	8	8	2	3	24	4	3	0	0	26	4	3
0	1	22	5	5	3	4	22	6	-4	-4	0	24	4	5	3	3	24	4	-2	-2	1	26	7	-4
3	1	22	18	15	4	4	22	4	-4	-2	0	24	13	-15	-2	4	24	7	5	-1	1	26	4	-1
4	1	22	17	-15	0	5	22	6	7	-1	0	24	4	3	0	4	24	4	-1	0	1	26	4	2
6	1	22	9	8	2	5	22	6	-8	0	0	24	8	10	1	4	24	4	4	2	1	26	4	6
-7	2	22	4	-2	-3	1	23	7	7	1	0	24	8	-7	-3	1	25	5	4	3	1	26	5	-4
-5	2	22	4	3	-2	1	23	4	3	2	0	24	6	-9	-2	1	25	6	-6	0	3	26	4	-3
-4	2	22	6	6	2	1	23	7	-6	4	0	24	9	10	0	1	25	4	4	1	3	26	6	-6
-3	2	22	5	-3	5	1	23	16	-17	-6	1	24	7	-7	1	1	25	5	-6	0	1	27	4	-4
-2	2	22	8	-8	6	1	23	17	18															

DWARFA

STRUCTURE (II)

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	9	-8	0	8	0	64	-65	5	2	1	10	-10	5	7	1	12	-11	-3	13	1	10	11
3	0	0	42	-42	1	8	0	37	-35	6	2	1	9	-11	6	7	1	8	6	-1	13	1	9	-8
4	0	0	27	-28	3	8	0	7	7	-4	3	1	21	-21	-5	8	1	10	-11	0	13	1	13	-12
5	0	0	15	15	4	8	0	16	13	-3	3	1	10	-10	-4	8	1	14	-13	1	13	1	8	6
1	1	0	16	17	2	9	0	12	12	-1	3	1	33	34	-3	8	1	12	10	2	13	1	11	11
2	1	0	17	-17	3	9	0	14	14	0	3	1	55	55	-2	8	1	16	18	5	13	1	7	-4
3	1	0	8	8	0	10	0	20	-18	1	3	1	59	-59	0	8	1	15	-15	1	14	1	6	-7
5	1	0	10	-10	2	10	0	10	8	2	3	1	24	-25	1	8	1	16	16	2	14	1	8	-8
1	2	0	10	11	3	10	0	21	21	5	3	1	6	7	2	8	1	21	20	3	14	1	7	-4
2	2	0	6	3	5	10	0	7	-7	-3	4	1	20	20	3	8	1	9	10	-1	15	1	7	-7
3	2	0	32	-31	1	11	0	8	7	-1	4	1	46	-46	4	8	1	13	13	1	15	1	10	10
4	2	0	10	-8	0	12	0	9	-9	0	4	1	118	-119	5	8	1	9	-11	2	15	1	7	7
5	2	0	18	19	2	12	0	12	13	1	4	1	37	-37	-4	9	1	12	10	-5	0	2	6	-5
6	2	0	10	9	3	12	0	14	13	3	4	1	13	14	-1	9	1	23	-23	-3	0	2	56	56
1	3	0	47	-49	2	14	0	6	7	4	4	1	6	5	0	9	1	25	-25	-2	0	2	54	54
2	3	0	27	-26	3	14	0	10	11	5	4	1	11	-11	1	9	1	14	15	-1	0	2	67	-67
4	3	0	14	14	0	16	0	7	-6	-5	5	1	9	7	2	9	1	25	24	0	0	2	92	-94
0	4	0	29	-30	-5	1	1	18	-18	-2	5	1	8	-8	3	9	1	17	17	1	0	2	49	-49
1	4	0	15	14	-4	1	1	33	-32	-1	5	1	28	-28	5	9	1	8	-6	2	0	2	29	28
2	4	0	22	-22	-2	1	1	51	50	1	5	1	27	27	6	9	1	8	-4	3	0	2	41	40
3	4	0	11	-11	-1	1	1	116	119	-5	6	1	22	-21	-6	10	1	7	1	4	0	2	16	16
5	4	0	6	-1	1	1	1	89	-90	-3	6	1	11	10	-4	10	1	8	-8	5	0	2	12	-12
1	5	0	27	-28	2	1	1	31	-31	-2	6	1	31	30	-1	10	1	15	15	-5	1	2	8	-4
2	5	0	8	-8	3	1	1	36	-36	-1	6	1	31	30	3	10	1	11	10	-4	1	2	9	8
3	5	0	13	-13	4	1	1	32	33	0	6	1	25	-25	4	10	1	12	10	-3	1	2	16	15
4	5	0	22	23	5	1	1	15	15	1	6	1	9	-10	-4	11	1	16	15	-2	1	2	35	35
6	5	0	10	-9	-5	2	1	9	-7	2	6	1	21	19	-1	11	1	15	-16	-1	1	2	20	-19
0	6	0	71	-71	-3	2	1	33	32	3	6	1	17	18	1	11	1	16	16	0	1	2	47	-46
1	6	0	29	-29	-2	2	1	44	-44	5	6	1	15	-16	2	11	1	14	16	1	1	2	64	63
6	6	0	9	-9	-1	2	1	60	61	-4	7	1	14	14	3	11	1	7	4	2	1	2	19	-19
1	7	0	42	-42	1	2	1	38	-38	-2	7	1	33	-34	4	11	1	10	-9	-5	2	2	15	-14
3	7	0	13	16	2	2	1	13	-13	-1	7	1	45	-42	5	11	1	7	-8	-3	2	2	40	39
4	7	0	23	23	3	2	1	16	-15	1	7	1	35	36	0	12	1	11	11	-2	2	2	77	78
6	7	0	7	-8	4	2	1	7	-5	4	7	1	9	-8	-4	13	1	9	11	-1	2	2	72	73

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	2	2	45	-47	-2	6	2	18	-17	1	10	2	28	28	2	1	3	23	23	-1	5	3	10	-9
1	2	2	41	-42	1	6	2	26	-27	3	10	2	7	-4	3	1	3	10	10	0	5	3	44	-43
2	2	2	14	-13	5	6	2	16	17	4	10	2	11	-11	4	1	3	7	-7	1	5	3	24	-23
4	2	2	17	17	-6	7	2	9	-8	-4	11	2	9	-9	5	1	3	12	-14	2	5	3	12	11
5	2	2	6	-9	-5	7	2	8	-9	-3	11	2	6	4	-4	2	3	10	-10	3	5	3	14	13
6	2	2	10	-9	-4	7	2	11	11	0	11	2	9	-8	-2	2	3	19	-19	4	5	3	12	13
-4	3	2	21	21	-3	7	2	15	15	2	11	2	10	10	-1	2	3	56	-55	-4	6	3	18	17
-3	3	2	6	-6	-2	7	2	7	3	3	11	2	11	10	0	2	3	44	-41	-3	6	3	19	-20
-1	3	2	52	51	-1	7	2	17	-17	-5	12	2	11	11	1	2	3	20	-20	-2	6	3	20	-19
0	3	2	31	-31	0	7	2	6	-5	-4	12	2	9	10	2	2	3	10	-7	-1	6	3	17	-18
1	3	2	39	40	1	7	2	26	27	-3	12	2	8	-5	3	2	3	7	5	0	6	3	13	11
2	3	2	19	-18	2	7	2	10	9	-2	12	2	9	-8	4	2	3	12	-12	1	6	3	11	12
3	3	2	9	-10	4	7	2	11	-14	-1	12	2	10	-9	5	2	3	7	5	2	6	3	7	-8
4	3	2	14	-15	5	7	2	9	-9	0	12	2	8	10	-6	3	3	8	-5	4	6	3	13	-12
5	3	2	10	-11	-3	8	2	13	-15	1	12	2	24	25	-5	3	3	7	-9	5	6	3	8	7
-6	4	2	8	4	-2	8	2	9	-10	3	12	2	8	-8	-4	3	3	13	13	6	6	3	9	8
-5	4	2	9	-9	-1	8	2	7	-8	4	12	2	10	-12	-3	3	3	28	28	-4	7	3	12	-13
-4	4	2	6	-6	0	8	2	33	33	-1	13	2	7	5	-2	3	3	25	24	-2	7	3	14	-14
-2	4	2	29	-29	1	8	2	21	22	1	13	2	13	-13	-1	3	3	46	-45	3	7	3	9	7
-1	4	2	25	26	2	8	2	7	5	3	13	2	7	-3	0	3	3	19	-20	-5	8	3	7	6
1	4	2	19	-19	3	8	2	9	-10	-2	14	2	9	-7	2	3	3	14	13	-4	8	3	14	15
2	4	2	13	-15	4	8	2	13	-15	0	14	2	16	17	3	3	3	18	18	-1	8	3	8	-8
4	4	2	16	17	-5	9	2	9	-10	1	14	2	12	12	-6	4	3	10	-12	1	8	3	27	27
-4	5	2	13	14	-3	9	2	14	14	3	14	2	7	-7	-3	4	3	8	7	2	8	3	20	20
-3	5	2	11	11	-1	9	2	14	-14	4	14	2	10	-8	-2	4	3	23	23	4	8	3	6	-4
-1	5	2	11	-12	0	9	2	15	-15	-3	15	2	7	-5	-1	4	3	27	-26	-4	9	3	8	-8
0	5	2	26	-27	2	9	2	8	10	0	15	2	7	3	2	4	3	54	-53	-3	9	3	8	-10
1	5	2	31	32	3	9	2	16	17	0	17	2	13	9	3	4	3	13	-12	-1	9	3	15	14
2	5	2	24	23	4	9	2	8	-7	-4	1	3	26	26	4	4	3	17	-16	0	9	3	26	24
4	5	2	10	-8	-5	10	2	8	7	-3	1	3	30	30	6	4	3	8	7	2	9	3	16	-16
5	5	2	14	-14	-3	10	2	10	-8	-2	1	3	11	-10	-5	5	3	7	-6	3	9	3	19	-20
7	5	2	9	6	-2	10	2	14	-15	-1	1	3	19	17	-4	5	3	9	-9	4	9	3	9	-9
-4	6	2	17	-17	-1	10	2	20	-19	0	1	3	44	-44	-3	5	3	17	-16	5	9	3	10	12
-3	6	2	24	-23	0	10	2	15	14	1	1	3	23	23	-2	5	3	14	13	-4	10	3	12	13

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	10	3	9	-9	0	1	4	20	-20	1	5	4	24	-22	1	10	4	21	-22	-1	2	5	13	14
-1	10	3	8	4	1	1	4	13	-13	2	5	4	12	-12	2	10	4	22	-23	0	2	5	25	26
0	10	3	15	17	2	1	4	12	-11	3	5	4	14	-14	3	10	4	11	-12	1	2	5	15	-16
1	10	3	25	25	-4	2	4	11	11	5	5	4	14	14	4	10	4	10	10	2	2	5	8	-7
2	10	3	16	17	-3	2	4	22	-21	-3	6	4	7	7	0	11	4	12	12	3	2	5	10	-11
4	10	3	10	-11	-2	2	4	19	-19	-1	6	4	7	3	1	11	4	10	9	4	2	5	10	9
-3	11	3	10	-9	-1	2	4	25	-25	0	6	4	35	35	2	11	4	8	-6	-5	3	5	8	10
-2	11	3	8	-8	0	2	4	38	-37	1	6	4	14	15	3	11	4	10	-10	-3	3	5	18	-20
-1	11	3	7	8	1	2	4	30	30	2	6	4	13	11	-4	12	4	8	-8	-2	3	5	13	-13
0	11	3	39	38	2	2	4	15	14	4	6	4	8	-8	-2	12	4	14	15	-1	3	5	14	-13
2	11	3	10	-10	4	2	4	12	-11	5	6	4	7	2	-1	12	4	21	22	0	3	5	35	34
3	11	3	15	-14	5	2	4	6	-5	-5	7	4	10	11	1	12	4	23	-22	1	3	5	21	21
5	11	3	7	6	-5	3	4	10	11	-3	7	4	15	-16	2	12	4	18	-19	4	3	5	13	-14
-1	12	3	10	-12	-4	3	4	15	16	0	7	4	24	25	3	12	4	9	-8	-6	4	5	8	9
0	12	3	8	-11	-3	3	4	6	-5	1	7	4	14	14	4	12	4	9	7	-5	4	5	8	8
2	12	3	7	8	-2	3	4	41	-42	2	7	4	6	-8	1	14	4	13	-11	-4	4	5	19	-19
-4	13	3	7	-6	-1	3	4	26	-25	-4	8	4	10	-10	2	14	4	9	-8	-3	4	5	28	-29
-3	13	3	9	-10	1	3	4	14	-12	-3	8	4	7	-6	-3	15	4	8	4	-2	4	5	15	-14
-1	13	3	22	21	4	3	4	10	-10	-1	8	4	11	11	0	15	4	13	-14	1	4	5	12	11
0	13	3	22	23	5	3	4	11	11	0	8	4	7	8	3	15	4	7	3	4	4	5	17	17
2	13	3	12	-11	-4	4	4	15	15	1	8	4	7	-8	0	17	4	8	-8	5	4	5	10	7
3	13	3	9	-13	-3	4	4	20	20	2	8	4	8	5	-5	1	5	15	15	-3	5	5	17	-19
-1	16	3	10	8	-2	4	4	17	-16	3	8	4	8	5	-4	1	5	7	7	-2	5	5	10	-9
1	16	3	13	-13	-1	4	4	39	-37	4	8	4	11	11	-2	1	5	16	-15	-1	5	5	16	15
-6	0	4	9	-8	0	4	4	12	-12	5	8	4	11	10	-1	1	5	24	-24	0	5	5	20	20
-4	0	4	28	29	1	4	4	29	28	-3	9	4	11	-11	0	1	5	15	15	1	5	5	17	17
-3	0	4	7	6	2	4	4	32	31	-2	9	4	9	6	1	1	5	23	22	2	5	5	6	7
-1	0	4	33	-33	3	4	4	10	10	-1	9	4	15	14	2	1	5	7	-7	3	5	5	16	-16
0	0	4	22	-23	4	4	4	7	-6	0	9	4	27	28	3	1	5	13	-13	5	5	5	9	9
2	0	4	37	-37	5	4	4	11	-9	1	9	4	16	16	4	1	5	20	-20	-4	6	5	6	-1
4	0	4	19	-19	-5	5	4	12	13	2	9	4	12	-11	6	1	5	8	5	-2	6	5	9	-10
5	0	4	10	-12	-3	5	4	12	12	-4	10	4	12	-11	-5	2	5	8	9	-1	6	5	12	10
-3	1	4	10	-12	-2	5	4	11	-12	-2	10	4	14	12	-3	2	5	10	-10	0	6	5	10	12
-1	1	4	15	-16	-1	5	4	21	-20	-1	10	4	30	31	-2	2	5	9	-10	1	6	5	7	-9

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	6	5	11	-9	0	13	5	11	-11	2	4	6	18	-17	-1	12	6	11	-10	3	5	7	7	7
-5	7	5	10	-11	1	13	5	10	-11	4	4	6	12	12	0	12	6	15	-16	-5	6	7	10	-10
-3	7	5	6	4	-1	14	5	7	-7	5	4	6	7	1	2	12	6	10	9	-4	6	7	7	-1
-1	7	5	9	8	-2	16	5	8	-4	-5	5	6	7	-7	1	14	6	7	-2	-2	6	7	9	8
0	7	5	10	-9	-4	0	6	18	-18	-4	5	6	21	-23	-4	1	7	16	-15	0	6	7	14	-14
4	7	5	16	16	-3	0	6	18	-18	-2	5	6	6	4	-3	1	7	7	-8	2	6	7	9	8
-4	8	5	12	-12	-2	0	6	15	-15	-1	5	6	19	19	-2	1	7	10	10	3	6	7	16	15
-2	8	5	8	8	0	0	6	36	35	1	5	6	20	-21	-1	1	7	22	22	0	7	7	12	14
-1	8	5	17	16	2	0	6	15	-15	4	5	6	8	8	0	1	7	13	-14	1	7	7	14	15
0	8	5	14	14	3	0	6	17	-16	-2	6	6	6	9	1	1	7	33	-34	2	7	7	11	11
1	8	5	15	-16	5	0	6	16	15	-1	6	6	9	-9	2	1	7	23	-23	4	7	7	7	-5
2	8	5	10	-9	6	0	6	7	4	1	6	6	10	9	4	1	7	18	18	-2	8	7	7	5
3	8	5	8	-6	-4	1	6	11	-8	3	6	6	16	17	-5	2	7	9	-7	0	8	7	6	-5
5	8	5	8	7	-2	1	6	10	10	-1	7	6	12	11	-3	2	7	7	6	-5	9	7	8	8
-5	9	5	7	-8	2	1	6	9	-10	0	7	6	11	-11	-1	2	7	8	-9	-4	9	7	12	10
-4	9	5	8	-7	-5	2	6	7	6	4	7	6	9	9	0	2	7	12	-11	-3	9	7	8	-8
-3	9	5	7	5	-3	2	6	14	-14	-4	8	6	14	15	1	2	7	7	5	-2	9	7	12	-13
-2	9	5	15	15	-2	2	6	13	-13	-3	8	6	13	13	2	2	7	27	28	-1	9	7	15	-15
-1	9	5	11	12	-1	2	6	25	25	-2	8	6	11	11	3	2	7	10	9	0	9	7	6	3
0	9	5	16	-16	0	2	6	27	26	-1	8	6	12	-12	-4	3	7	8	-7	1	9	7	15	17
1	9	5	13	-13	2	2	6	19	-19	0	8	6	16	-16	-3	3	7	8	9	2	9	7	8	8
3	9	5	11	12	3	2	6	27	-27	2	8	6	7	7	-2	3	7	15	15	-3	10	7	9	10
1	10	5	11	-10	5	2	6	10	9	3	8	6	8	7	-1	3	7	7	7	-2	11	7	7	-7
3	10	5	7	4	-5	3	6	12	-14	-2	9	6	7	8	1	3	7	19	-19	-1	11	7	14	-11
-3	11	5	18	19	-4	3	6	16	-16	-1	9	6	9	10	2	3	7	18	-17	1	11	7	11	10
-2	11	5	17	18	0	3	6	13	-14	-4	10	6	11	9	5	3	7	7	5	-2	13	7	7	-6
-1	11	5	7	-6	1	3	6	29	-30	-3	10	6	11	10	-4	4	7	9	11	-1	13	7	7	-9
0	11	5	22	-24	3	3	6	16	17	-2	10	6	8	-8	-3	4	7	11	10	2	13	7	9	4
1	11	5	23	-24	4	3	6	9	10	-1	10	6	15	-16	-2	4	7	13	12	-3	0	8	18	18
3	11	5	13	13	-4	4	6	11	-11	0	10	6	16	-17	0	4	7	24	-23	-2	0	8	18	18
-2	12	5	11	12	-3	4	6	7	-4	2	10	6	15	17	2	4	7	8	7	-1	0	8	8	-9
-1	12	5	8	9	-2	4	6	8	8	3	10	6	7	8	3	4	7	7	3	0	0	8	22	-24
2	12	5	10	-12	-1	4	6	24	23	-2	11	6	9	12	-1	5	7	9	8	1	0	8	27	-27
-3	13	5	7	7	0	4	6	13	14	-1	11	6	7	1	2	5	7	11	15	3	0	8	20	20

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	8	9	9	-4	5	8	8	7	-4	9	8	8	6	-1	3	9	8	-7	0	10	9	7	1
-3	1	8	9	-10	-1	5	8	17	-18	0	9	8	11	-10	1	3	9	8	7	-2	0	10	9	-10
1	1	8	19	19	1	5	8	14	13	-3	10	8	8	-6	-1	4	9	7	1	-1	0	10	10	-12
-3	2	8	12	11	2	5	8	12	12	-2	10	8	8	-6	0	4	9	12	10	0	0	10	14	13
0	2	8	22	-23	-1	7	8	8	-5	0	10	8	8	10	1	4	9	11	11	1	0	10	22	23
1	2	8	12	-11	0	7	8	7	-8	2	11	8	8	-2	-4	5	9	7	3	0	1	10	8	-7
2	2	8	15	16	2	7	8	8	6	-2	12	8	8	-6	-4	6	9	8	8	-2	2	10	8	-8
3	2	8	18	16	-4	8	8	8	-8	-2	1	9	11	-11	-2	6	9	8	-7	-1	2	10	9	-8
-4	3	8	8	5	-3	8	8	12	-13	-1	1	9	18	-18	-1	8	9	8	-9	0	5	10	9	7
-1	3	8	7	-7	-1	8	8	7	8	0	1	9	17	-17	0	8	9	7	1	-1	6	10	7	4
0	3	8	7	6	0	8	8	16	17	1	1	9	9	8	1	8	9	8	7	-2	7	10	9	-6
1	3	8	16	16	1	8	8	7	8	2	1	9	19	19	-2	9	9	9	7	0	1	11	13	12
0	4	8	9	-6	2	8	8	8	-2	0	2	9	8	7	-1	9	9	8	9					

DWARFB

APPENDIX EIGHT

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	10	9	2	5	0	27	-27	4	10	0	6	-6	-7	2	1	4	1	1	4	1	21	-23
3	0	0	32	-32	3	5	0	39	-41	5	10	0	5	-4	-6	2	1	9	-9	2	4	1	27	-26
4	0	0	30	-28	4	5	0	29	-30	6	10	0	6	-7	-5	2	1	6	-6	3	4	1	6	-6
5	0	0	15	-15	5	5	0	8	-8	1	11	0	20	-20	-4	2	1	5	7	4	4	1	5	2
6	0	0	43	-43	6	5	0	11	9	2	11	0	23	-24	-3	2	1	38	38	5	4	1	29	29
7	0	0	32	-33	7	5	0	6	6	3	11	0	31	-31	-2	2	1	18	-18	6	4	1	17	18
8	0	0	11	-10	1	6	0	15	-13	4	11	0	20	-19	-1	2	1	25	23	7	4	1	6	5
2	1	0	50	-53	2	6	0	17	17	5	11	0	6	-6	0	2	1	6	8	8	4	1	5	3
3	1	0	57	-53	3	6	0	13	-15	0	12	0	33	-33	2	2	1	6	-8	-8	5	1	9	8
4	1	0	14	-14	4	6	0	5	5	1	12	0	25	-24	3	2	1	27	-29	-7	5	1	18	17
5	1	0	12	-12	6	6	0	8	-7	3	12	0	6	6	4	2	1	8	7	-6	5	1	6	-6
6	1	0	6	5	7	6	0	7	9	4	12	0	9	8	5	2	1	7	7	-5	5	1	4	4
7	1	0	10	7	8	6	0	5	-4	5	12	0	6	6	7	2	1	10	-9	-4	5	1	19	-18
8	1	0	13	12	1	7	0	33	32	1	13	0	6	6	8	2	1	4	2	-3	5	1	33	-34
1	2	0	72	-74	3	7	0	29	-29	2	13	0	26	25	-8	3	1	10	-8	-2	5	1	16	-20
2	2	0	33	-33	5	7	0	6	-6	3	13	0	8	7	-7	3	1	19	-18	-1	5	1	38	-39
3	2	0	4	-2	6	7	0	26	28	4	13	0	9	10	-6	3	1	14	-15	0	5	1	9	-10
5	2	0	27	28	7	7	0	7	6	0	14	0	9	8	-4	3	1	15	13	1	5	1	8	-10
6	2	0	22	22	0	8	0	22	-22	1	14	0	17	17	-3	3	1	24	23	2	5	1	22	21
7	2	0	8	9	1	8	0	21	-21	2	14	0	10	10	-2	3	1	13	14	3	5	1	9	10
1	3	0	18	-17	2	8	0	17	-17	3	14	0	5	4	-1	3	1	97	99	4	5	1	29	30
2	3	0	47	48	3	8	0	4	-2	-9	1	1	5	4	0	3	1	53	54	7	5	1	8	11
3	3	0	80	80	4	8	0	19	18	-8	1	1	6	-7	2	3	1	23	-24	-7	6	1	19	-21
4	3	0	49	51	5	8	0	22	23	-7	1	1	9	8	3	3	1	5	-5	-6	6	1	6	-7
5	3	0	10	9	6	8	0	12	12	-6	1	1	7	6	4	3	1	7	5	-4	6	1	34	-36
0	4	0	4	4	7	8	0	5	5	-5	1	1	20	-20	5	3	1	13	-12	-3	6	1	10	-13
1	4	0	21	21	1	9	0	12	12	-4	1	1	55	-49	6	3	1	17	-18	-1	6	1	26	25
2	4	0	41	41	2	9	0	16	16	-1	1	1	13	13	-7	4	1	6	8	0	6	1	30	29
3	4	0	5	1	3	9	0	23	25	1	1	1	64	-65	-6	4	1	19	19	1	6	1	12	16
4	4	0	19	-21	4	9	0	19	19	2	1	1	53	53	-5	4	1	12	11	2	6	1	8	9
5	4	0	16	-18	7	9	0	5	-3	3	1	1	18	-16	-4	4	1	21	20	3	6	1	28	32
6	4	0	9	-11	0	10	0	16	16	4	1	1	4	-2	-2	4	1	47	-43	4	6	1	5	5
7	4	0	6	4	1	10	0	6	7	5	1	1	18	16	-1	4	1	90	-87	6	6	1	12	13
1	5	0	20	19	3	10	0	5	-5	6	1	1	9	9	0	4	1	44	-43	7	6	1	5	-3

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-8	7	1	4	-5	4	9	1	10	10	-1	15	1	8	-8	7	1	2	12	13	0	4	2	64	-60
-5	7	1	21	21	5	9	1	6	8	0	15	1	7	-8	8	1	2	6	7	2	4	2	22	-22
-4	7	1	22	23	6	9	1	5	7	1	15	1	10	10	-8	2	2	9	7	3	4	2	33	-31
-3	7	1	24	26	-6	10	1	4	-5	2	15	1	5	5	-7	2	2	7	7	4	4	2	13	-15
-2	7	1	22	23	-5	10	1	9	-9	-8	0	2	17	-16	-5	2	2	23	-22	5	4	2	8	-9
-1	7	1	6	-5	-4	10	1	7	-5	-7	0	2	10	-9	-4	2	2	18	-19	6	4	2	14	12
1	7	1	17	-16	-3	10	1	6	-7	-6	0	2	7	-6	-3	2	2	5	-6	7	4	2	9	7
2	7	1	26	-25	-1	10	1	16	16	-5	0	2	8	7	-2	2	2	41	-41	-6	5	2	20	23
3	7	1	33	-34	0	10	1	23	23	-4	0	2	8	9	0	2	2	73	-69	-5	5	2	14	13
4	7	1	9	-8	1	10	1	22	22	-3	0	2	13	12	1	2	2	15	14	-4	5	2	22	24
5	7	1	10	-12	2	10	1	7	7	-2	0	2	6	8	2	2	2	38	38	-3	5	2	15	-13
-6	8	1	11	11	3	10	1	9	8	-1	0	2	114	125	3	2	2	28	27	-2	5	2	29	-30
-5	8	1	35	36	6	10	1	6	-6	0	0	2	29	27	4	2	2	32	34	1	5	2	33	-32
-4	8	1	20	18	-5	11	1	9	10	1	0	2	101	-104	5	2	2	13	13	2	5	2	27	-27
-3	8	1	25	26	-4	11	1	4	4	2	0	2	34	-34	8	2	2	5	-2	4	5	2	4	4
-2	8	1	16	-15	-3	11	1	7	8	3	0	2	26	-28	-7	3	2	4	4	5	5	2	15	16
-1	8	1	19	-19	-2	11	1	11	11	4	0	2	20	19	-6	3	2	11	10	6	5	2	5	-4
0	8	1	14	-14	-1	11	1	4	3	5	0	2	12	-11	-5	3	2	10	-10	-8	6	2	4	-4
1	8	1	22	-24	0	11	1	11	9	6	0	2	12	-12	-3	3	2	5	-5	-7	6	2	5	-4
2	8	1	44	-44	4	11	1	7	-6	7	0	2	6	5	-2	3	2	32	31	-6	6	2	15	-15
3	8	1	21	-22	5	11	1	8	-8	8	0	2	16	16	-1	3	2	58	55	-4	6	2	16	-16
4	8	1	7	-7	-5	12	1	5	-2	-8	1	2	6	6	0	3	2	42	-39	-3	6	2	11	11
5	8	1	4	-1	-4	12	1	6	-5	-7	1	2	15	16	1	3	2	38	-33	-2	6	2	36	34
6	8	1	16	17	-1	12	1	11	12	-6	1	2	11	11	2	3	2	23	23	-1	6	2	23	25
-7	9	1	5	5	0	12	1	11	-12	-5	1	2	5	4	3	3	2	15	16	0	6	2	27	26
-6	9	1	10	9	4	12	1	5	3	-3	1	2	15	13	4	3	2	4	-4	1	6	2	21	19
-4	9	1	16	-16	-2	13	1	4	-3	-2	1	2	54	-51	5	3	2	15	-16	2	6	2	20	20
-3	9	1	15	-14	-1	13	1	14	-15	-1	1	2	44	-45	6	3	2	16	-16	5	6	2	21	-21
-2	9	1	25	-24	0	13	1	9	-9	0	1	2	114	-116	-8	4	2	6	7	-7	7	2	5	-5
-1	9	1	19	-18	-4	14	1	6	5	1	1	2	49	-51	-6	4	2	5	-5	-6	7	2	15	16
0	9	1	10	-8	-3	14	1	7	6	3	1	2	8	10	-5	4	2	10	9	-5	7	2	16	17
1	9	1	5	7	-1	14	1	7	8	4	1	2	27	27	-4	4	2	18	19	-4	7	2	7	8
2	9	1	9	10	1	14	1	8	8	5	1	2	21	21	-3	4	2	37	37	-3	7	2	15	16
3	9	1	16	17	3	14	1	11	-10	6	1	2	21	22	-1	4	2	34	-34	-2	7	2	32	-31

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-1	7	2	5	-6	-3	10	2	18	19	0	13	2	16	16	-9	3	3	10	-8	-3	5	3	12	10
1	7	2	16	-15	-1	10	2	17	17	1	13	2	20	20	-7	3	3	12	-10	-2	5	3	10	8
2	7	2	29	-29	0	10	2	17	17	-4	14	2	7	8	-6	3	3	14	15	-1	5	3	13	13
3	7	2	5	-6	1	10	2	10	-10	-2	14	2	6	5	-5	3	3	17	17	0	5	3	30	26
4	7	2	8	7	2	10	2	25	-24	2	14	2	8	-8	-4	3	3	26	26	1	5	3	43	42
5	7	2	22	21	3	10	2	20	-21	3	14	2	6	-4	-3	3	3	7	6	2	5	3	12	12
6	7	2	12	11	4	10	2	16	-15	-2	15	2	6	-6	-2	3	3	10	-8	3	5	3	6	7
7	7	2	8	-9	5	10	2	4	-6	0	15	2	5	-3	-1	3	3	11	-10	4	5	3	7	-7
-7	8	2	6	6	6	10	2	5	-5	-8	1	3	6	5	0	3	3	60	-58	5	5	3	16	-15
-6	8	2	18	20	-6	11	2	7	6	-7	1	3	10	9	1	3	3	13	12	6	5	3	5	4
-4	8	2	17	-17	-4	11	2	15	15	-6	1	3	4	1	2	3	3	13	-14	7	5	3	6	-5
-3	8	2	11	-13	-3	11	2	9	9	-4	1	3	15	14	3	3	3	22	21	-8	6	3	10	-11
-2	8	2	15	-14	-2	11	2	8	7	-3	1	3	14	13	6	3	3	10	10	-6	6	3	9	8
0	8	2	12	-12	-1	11	2	6	-5	-2	1	3	7	-8	7	3	3	4	5	-5	6	3	5	-5
1	8	2	6	-5	0	11	2	24	-24	-1	1	3	32	-29	-8	4	3	13	13	-4	6	3	9	9
2	8	2	8	8	1	11	2	16	-15	0	1	3	81	-77	-7	4	3	26	25	-3	6	3	18	19
3	8	2	25	26	2	11	2	16	-16	1	1	3	4	4	-6	4	3	8	8	-2	6	3	24	23
4	8	2	12	11	3	11	2	4	-3	2	1	3	5	-4	-5	4	3	15	-15	-1	6	3	18	18
-5	9	2	21	-22	4	11	2	4	3	3	1	3	9	-10	-4	4	3	9	8	0	6	3	13	-12
-4	9	2	26	-28	5	11	2	6	6	4	1	3	7	7	-3	4	3	42	-41	1	6	3	15	-13
-3	9	2	11	-12	-4	12	2	9	-9	6	1	3	7	8	-2	4	3	29	-29	2	6	3	4	-2
-2	9	2	4	4	-3	12	2	12	-11	-9	2	3	9	-8	-1	4	3	57	-59	3	6	3	9	-8
-1	9	2	12	11	-2	12	2	25	-25	-8	2	3	6	-5	0	4	3	23	-24	4	6	3	17	-16
0	9	2	11	13	-1	12	2	21	-22	-6	2	3	8	-6	1	4	3	27	28	6	6	3	6	-7
1	9	2	21	21	0	12	2	10	9	-5	2	3	12	12	2	4	3	29	29	-6	7	3	9	9
2	9	2	20	22	1	12	2	16	17	-4	2	3	36	36	3	4	3	14	14	-5	7	3	18	18
3	9	2	20	20	2	12	2	20	22	-3	2	3	37	35	4	4	3	18	16	-4	7	3	22	21
4	9	2	6	-6	3	12	2	10	11	-2	2	3	41	42	5	4	3	12	13	-3	7	3	7	8
5	9	2	5	-5	4	12	2	7	7	-1	2	3	47	48	6	4	3	7	-8	-2	7	3	23	-22
6	9	2	9	-8	-5	13	2	7	-7	0	2	3	39	-36	-8	5	3	6	5	-1	7	3	12	-12
-7	10	2	4	-5	-4	13	2	6	-6	2	2	3	18	-19	-7	5	3	11	-12	0	7	3	8	-7
-6	10	2	5	4	-3	13	2	7	-7	3	2	3	15	-17	-6	5	3	22	-22	1	7	3	20	-20
-5	10	2	6	5	-2	13	2	7	5	5	2	3	21	-21	-5	5	3	15	-15	2	7	3	29	-30
-4	10	2	10	9	-1	13	2	26	27	6	2	3	14	-13	-4	5	3	21	-21	3	7	3	11	-11

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	7	3	5	4	-4	11	3	12	-12	-8	1	4	15	13	5	3	4	22	-24	3	6	4	5	4
5	7	3	6	3	-1	11	3	14	14	-6	1	4	12	-12	-7	4	4	11	-12	4	6	4	4	-5
6	7	3	4	1	2	11	3	15	14	-4	1	4	6	5	-6	4	4	13	15	6	6	4	7	7
-7	8	3	5	5	4	11	3	7	-6	-2	1	4	36	-39	-5	4	4	22	21	-8	7	4	5	3
-6	8	3	13	14	-5	12	3	13	-13	-1	1	4	42	-44	-4	4	4	17	17	-7	7	4	5	6
-4	8	3	7	-6	-4	12	3	5	-4	0	1	4	33	-26	-3	4	4	10	-9	-5	7	4	7	-8
-3	8	3	14	-14	-2	12	3	12	12	1	1	4	23	24	-2	4	4	25	-22	-4	7	4	11	-9
-2	8	3	15	-15	0	12	3	7	-8	3	1	4	11	11	-1	4	4	13	-14	-3	7	4	9	-9
-1	8	3	7	8	1	12	3	6	6	4	1	4	15	17	0	4	4	13	12	-2	7	4	14	-14
1	8	3	8	-7	3	12	3	6	4	5	1	4	8	9	2	4	4	38	-38	1	7	4	7	6
2	8	3	4	4	-5	13	3	10	9	6	1	4	9	9	3	4	4	5	-4	2	7	4	9	9
3	8	3	16	17	-4	13	3	8	7	-8	2	4	6	6	4	4	4	8	-9	3	7	4	9	9
4	8	3	5	6	-1	13	3	5	-5	-6	2	4	10	10	5	4	4	9	10	4	7	4	7	9
5	8	3	4	3	0	13	3	5	-4	-4	2	4	17	15	6	4	4	9	9	5	7	4	6	5
6	8	3	5	-5	1	13	3	10	-10	-3	2	4	17	-18	-8	5	4	6	-5	-6	8	4	7	-6
-7	9	3	6	-8	2	13	3	7	-8	-2	2	4	27	-26	-7	5	4	4	4	-5	8	4	30	-31
-6	9	3	4	-2	3	13	3	5	-5	-1	2	4	33	-30	-5	5	4	4	6	-4	8	4	7	-9
-5	9	3	26	-27	-2	14	3	5	-5	0	2	4	4	-2	-4	5	4	5	6	-2	8	4	21	20
-4	9	3	11	-12	-1	14	3	11	-10	1	2	4	25	26	-3	5	4	10	-10	-1	8	4	14	14
-2	9	3	5	5	1	14	3	5	5	2	2	4	4	-6	-2	5	4	45	42	0	8	4	5	-4
-1	9	3	36	36	0	15	3	10	11	3	2	4	14	13	-1	5	4	19	21	1	8	4	20	20
0	9	3	19	19	-9	0	4	5	-1	4	2	4	5	-5	0	5	4	11	10	2	8	4	23	23
1	9	3	18	18	-8	0	4	4	1	6	2	4	5	-5	1	5	4	4	-4	3	8	4	10	10
2	9	3	13	14	-7	0	4	6	8	7	2	4	7	-8	4	5	4	5	-6	4	8	4	7	-6
3	9	3	8	8	-6	0	4	4	6	-6	3	4	18	-18	5	5	4	16	-15	-7	9	4	5	3
4	9	3	5	-5	-5	0	4	16	15	-4	3	4	4	4	6	5	4	11	-11	-6	9	4	8	-7
-5	10	3	7	-6	-4	0	4	17	15	-3	3	4	10	-9	-7	6	4	7	-7	-5	9	4	11	-11
-4	10	3	5	4	-3	0	4	15	16	-2	3	4	31	33	-6	6	4	5	-5	-4	9	4	9	8
-3	10	3	8	-8	-2	0	4	15	14	-1	3	4	5	-7	-5	6	4	5	-4	-3	9	4	12	12
-2	10	3	12	11	-1	0	4	8	7	0	3	4	6	6	-4	6	4	13	-13	-2	9	4	8	8
1	10	3	6	-7	0	0	4	12	-12	1	3	4	41	40	-2	6	4	7	5	-1	9	4	11	-12
3	10	3	11	12	1	0	4	9	-11	2	3	4	15	12	0	6	4	8	7	0	9	4	5	6
5	10	3	5	-4	2	0	4	10	-7	3	3	4	6	-5	1	6	4	11	11	1	9	4	8	8
-5	11	3	9	-8	-9	1	4	5	2	4	3	4	15	-16	2	6	4	8	9	3	9	4	13	-13

OBSERVED AND CALCULATED STRUCTURE FACTORS :

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	9	4	14	-15	-8	1	5	5	-5	-6	4	5	30	-31	4	6	5	5	-5	-5	10	5	9	11
-7	10	4	5	4	-3	1	5	4	-3	-5	4	5	20	-20	5	6	5	5	3	-4	10	5	6	6
-4	10	4	16	16	-2	1	5	19	19	-4	4	5	32	-34	-8	7	5	8	7	-2	10	5	6	8
-1	10	4	7	7	-1	1	5	14	14	-3	4	5	32	-33	-7	7	5	11	11	-1	10	5	6	-5
0	10	4	19	-20	0	1	5	11	11	-2	4	5	17	-19	-6	7	5	14	14	3	10	5	7	-8
1	10	4	22	-22	1	1	5	6	-8	-1	4	5	25	-25	-5	7	5	19	19	4	10	5	7	-8
2	10	4	18	-18	2	1	5	4	-3	0	4	5	54	54	-4	7	5	5	4	-6	11	5	8	-8
3	10	4	9	-10	4	1	5	9	-10	1	4	5	44	46	-3	7	5	24	-24	-4	11	5	4	4
4	10	4	6	6	5	1	5	6	-5	2	4	5	20	20	-2	7	5	24	-24	-3	11	5	9	9
-6	11	4	6	-7	6	1	5	9	-9	3	4	5	8	8	-1	7	5	24	-24	-2	11	5	13	12
-4	11	4	5	7	-9	2	5	12	-9	4	4	5	4	3	0	7	5	23	-21	-1	11	5	7	-5
-3	11	4	7	-7	-8	2	5	5	-2	-8	5	5	10	-10	1	7	5	30	-29	0	11	5	15	-15
-2	11	4	8	-8	-7	2	5	12	-10	-7	5	5	15	-16	2	7	5	13	-13	3	11	5	9	-9
-1	11	4	26	-26	-5	2	5	18	17	-6	5	5	15	-15	3	7	5	10	11	-5	12	5	9	9
0	11	4	5	-5	-4	2	5	8	9	-5	5	5	10	-10	4	7	5	17	16	-4	12	5	7	7
1	11	4	5	4	-3	2	5	39	37	-4	5	5	5	-5	5	7	5	8	10	-2	12	5	9	-10
2	11	4	10	10	-2	2	5	37	39	-3	5	5	14	15	-5	8	5	8	-8	-1	12	5	18	-20
3	11	4	7	8	1	2	5	11	8	-2	5	5	16	15	-4	8	5	16	-16	0	12	5	8	-9
4	11	4	5	4	2	2	5	13	-12	-1	5	5	25	23	-3	8	5	12	-13	1	12	5	5	-5
-5	12	4	8	-8	3	2	5	8	-9	0	5	5	49	46	-2	8	5	5	-7	2	12	5	5	-3
-3	12	4	5	-5	4	2	5	17	-17	1	5	5	10	10	-1	8	5	11	11	-1	13	5	8	-9
-1	12	4	19	18	5	2	5	10	-11	2	5	5	9	-8	0	8	5	12	13	0	13	5	6	-6
0	12	4	20	19	6	2	5	9	9	3	5	5	14	-14	1	8	5	9	10	-9	0	6	9	10
1	12	4	13	14	-7	3	5	8	7	4	5	5	16	-16	2	8	5	16	15	-8	0	6	7	6
2	12	4	7	8	-6	3	5	25	25	-8	6	5	7	-7	3	8	5	7	6	-7	0	6	10	9
-4	13	4	5	5	-3	3	5	27	-29	-7	6	5	5	-4	5	8	5	5	-3	-6	0	6	24	-25
-3	13	4	6	5	-2	3	5	20	-20	-5	6	5	14	13	-7	9	5	7	-7	-5	0	6	17	-16
-2	13	4	13	12	-1	3	5	28	-27	-4	6	5	19	18	-6	9	5	16	-15	-4	0	6	18	-20
-1	13	4	6	7	0	3	5	8	8	-3	6	5	22	21	-5	9	5	17	-16	-3	0	6	22	-24
1	13	4	9	-8	2	3	5	8	8	-2	6	5	12	12	-3	9	5	4	-4	-2	0	6	21	-23
2	13	4	8	-7	3	3	5	18	18	0	6	5	8	6	-2	9	5	22	23	-1	0	6	15	-16
-1	14	4	5	-4	5	3	5	17	17	1	6	5	16	-16	-1	9	5	12	12	1	0	6	15	15
0	14	4	8	-7	-8	4	5	10	9	2	6	5	23	-22	1	9	5	15	15	2	0	6	25	26
1	14	4	7	-7	-7	4	5	4	-3	3	6	5	19	-18	2	9	5	5	6	3	0	6	9	9

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	6	16	17	-6	4	6	18	19	-3	8	6	12	10	-6	2	7	18	18	-1	5	7	10	10
-7	1	6	24	-24	-5	4	6	4	-1	-1	8	6	25	-25	-4	2	7	6	8	0	5	7	11	-11
-6	1	6	16	-16	-4	4	6	21	-21	0	8	6	7	-7	-3	2	7	12	12	1	5	7	33	-34
-5	1	6	12	-12	-3	4	6	26	-26	1	8	6	6	4	-2	2	7	18	-19	2	5	7	26	-25
-4	1	6	20	-21	-2	4	6	7	-7	3	8	6	6	-4	-1	2	7	22	-24	3	5	7	23	-22
-3	1	6	24	-25	0	4	6	8	-8	4	8	6	7	-4	0	2	7	14	-14	-7	6	7	16	15
-2	1	6	27	-28	1	4	6	32	-32	-5	9	6	10	8	1	2	7	4	-4	-6	6	7	19	19
-1	1	6	10	11	2	4	6	4	1	-3	9	6	4	-2	-6	3	7	17	-17	-5	6	7	9	9
0	1	6	32	33	3	4	6	7	9	-2	9	6	17	-15	-5	3	7	27	-27	-3	6	7	17	-16
1	1	6	23	23	4	4	6	6	7	1	9	6	9	-9	-4	3	7	27	-26	-2	6	7	28	-28
2	1	6	7	8	5	4	6	7	8	2	9	6	9	-9	-3	3	7	28	-29	-1	6	7	25	-23
3	1	6	15	15	-8	5	6	9	-8	3	9	6	10	-11	-2	3	7	10	-12	0	6	7	46	-44
4	1	6	7	8	-7	5	6	5	-5	-5	10	6	9	9	0	3	7	23	22	1	6	7	31	-31
-8	2	6	10	-9	-6	5	6	9	-9	-3	10	6	11	-11	1	3	7	22	23	3	6	7	14	14
-6	2	6	5	-5	-5	5	6	14	-12	-2	10	6	6	-6	2	3	7	26	26	4	6	7	21	21
-4	2	6	14	15	-4	5	6	20	-22	-1	10	6	18	-18	3	3	7	14	13	-7	7	7	4	5
-3	2	6	11	11	-3	5	6	7	7	0	10	6	11	-11	4	3	7	12	12	-5	7	7	7	-6
-2	2	6	36	36	-2	5	6	19	18	1	10	6	4	-4	-8	4	7	7	-8	-4	7	7	22	-23
-1	2	6	32	31	-1	5	6	18	17	3	10	6	7	7	-7	4	7	12	-12	-3	7	7	18	-19
0	2	6	30	26	0	5	6	18	17	-1	11	6	9	9	-6	4	7	16	-16	-2	7	7	18	-18
3	2	6	14	-13	2	5	6	13	12	0	11	6	21	20	-5	4	7	13	-12	-1	7	7	6	-6
4	2	6	12	-13	-8	6	6	5	-5	1	11	6	10	10	-4	4	7	7	-7	1	7	7	6	6
5	2	6	9	-10	-7	6	6	5	3	-4	12	6	5	5	-3	4	7	11	11	2	7	7	22	21
-7	3	6	6	-7	-4	6	6	12	-11	-3	12	6	6	7	-2	4	7	11	11	3	7	7	14	15
-6	3	6	10	9	-3	6	6	8	9	-2	12	6	8	9	-1	4	7	29	28	-7	8	7	11	-11
-5	3	6	23	23	-2	6	6	8	7	-1	12	6	7	8	0	4	7	26	25	-6	8	7	10	-10
-2	3	6	11	-10	-1	6	6	5	6	0	12	6	7	6	2	4	7	5	-3	-5	8	7	10	-10
-1	3	6	5	-6	0	6	6	11	10	-1	13	6	7	-6	3	4	7	10	-9	-4	8	7	5	3
0	3	6	5	5	2	6	6	7	7	-6	1	7	14	-15	-8	5	7	8	-7	-3	8	7	17	16
1	3	6	24	-24	-5	7	6	8	-8	-3	1	7	19	19	-6	5	7	6	6	-2	8	7	27	28
2	3	6	23	-23	-3	7	6	8	8	-2	1	7	25	26	-5	5	7	19	20	-1	8	7	38	39
3	3	6	8	-7	-1	7	6	11	10	0	1	7	17	-17	-4	5	7	30	30	0	8	7	9	9
5	3	6	5	5	0	7	6	4	-4	3	1	7	9	-10	-3	5	7	35	37	1	8	7	8	8
-7	4	6	10	10	-4	8	6	13	14	5	1	7	9	-10	-2	5	7	10	11	3	8	7	6	-7

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-6	9	7	10	9	-1	1	8	14	13	0	4	8	4	-3	-6	1	9	12	12	-1	5	9	8	-8
-5	9	7	17	18	0	1	8	9	-10	1	4	8	6	5	-5	1	9	11	12	0	5	9	21	-19
-4	9	7	16	16	1	1	8	22	-22	2	4	8	10	10	-3	1	9	8	8	1	5	9	10	-9
-3	9	7	12	14	2	1	8	18	-17	-7	5	8	6	6	-2	1	9	15	-15	2	5	9	5	-5
-2	9	7	7	8	3	1	8	6	-5	-5	5	8	8	8	-1	1	9	17	-16	-6	6	9	6	-5
-1	9	7	11	-12	4	1	8	5	-5	-4	5	8	18	19	0	1	9	7	-7	-5	6	9	6	-6
0	9	7	5	-6	-8	2	8	10	10	-3	5	8	19	20	2	1	9	8	-9	-4	6	9	9	-9
1	9	7	11	-10	-7	2	8	9	8	-2	5	8	11	11	-5	2	9	7	-6	-3	6	9	14	-15
2	9	7	9	-9	-6	2	8	9	9	0	5	8	12	-13	-3	2	9	6	-5	-2	6	9	7	-7
-4	10	7	5	-6	-5	2	8	20	20	3	5	8	5	-6	-2	2	9	10	-10	-1	6	9	16	-15
-2	10	7	10	-10	-4	2	8	8	9	-2	6	8	6	-7	0	2	9	12	11	1	6	9	18	19
-1	10	7	8	-7	-2	2	8	18	-19	-1	6	8	6	5	1	2	9	11	10	-5	7	9	5	-4
0	10	7	8	-9	-1	2	8	29	-30	0	6	8	7	-7	-7	3	9	9	-9	-4	7	9	6	5
-4	11	7	7	-7	0	2	8	21	-22	-4	7	8	12	13	-6	3	9	6	-5	-3	7	9	11	9
-2	11	7	8	-7	1	2	8	13	-12	-1	7	8	9	-10	-5	3	9	6	-5	-2	7	9	13	12
-1	11	7	5	-5	2	2	8	13	-11	0	7	8	18	-19	-2	3	9	9	8	-1	7	9	20	20
0	11	7	5	6	3	2	8	6	-4	1	7	8	5	6	-1	3	9	16	17	0	7	9	12	12
1	11	7	8	5	-7	3	8	6	5	-6	8	8	8	7	0	3	9	13	13	1	7	9	13	12
-7	0	8	34	-35	-6	3	8	9	10	-5	8	8	12	11	1	3	9	6	6	-5	8	9	13	12
-6	0	8	37	-37	-5	3	8	8	-8	-4	8	8	9	9	-7	4	9	6	5	-4	8	9	16	17
-5	0	8	5	-4	-4	3	8	22	-22	-2	8	8	11	-12	-6	4	9	7	7	-3	8	9	7	6
-4	0	8	11	-12	-3	3	8	26	-26	-1	8	8	5	-6	-5	4	9	15	15	-2	8	9	8	7
-3	0	8	6	7	-2	3	8	19	-18	2	8	8	5	-1	-4	4	9	16	16	-1	8	9	7	-7
-2	0	8	5	4	-1	3	8	6	-4	-5	9	8	7	-7	-3	4	9	12	13	0	8	9	7	-6
-1	0	8	24	23	0	3	8	11	-10	-4	9	8	10	-9	-2	4	9	5	4	-4	9	9	7	-7
0	0	8	36	38	1	3	8	6	-6	-3	9	8	11	-12	-1	4	9	5	-7	-3	9	9	12	-11
1	0	8	28	28	2	3	8	9	9	-2	9	8	6	-6	0	4	9	9	-10	-2	9	9	9	-10
-8	1	8	9	-8	3	3	8	13	14	-1	9	8	5	-4	1	4	9	12	-11	-6	0	10	19	20
-7	1	8	6	-6	-6	4	8	7	-7	0	9	8	8	-8	2	4	9	13	-12	-5	0	10	14	14
-6	1	8	19	19	-5	4	8	14	-14	-4	10	8	5	-5	-6	5	9	6	6	-4	0	10	12	11
-5	1	8	20	19	-4	4	8	5	-4	-3	10	8	4	1	-5	5	9	4	5	-3	0	10	9	9
-4	1	8	21	22	-3	4	8	14	14	-1	10	8	8	7	-4	5	9	4	5	-2	0	10	5	4
-3	1	8	13	15	-2	4	8	18	19	0	10	8	11	11	-3	5	9	9	-9	-1	0	10	14	14
-2	1	8	16	15	-1	4	8	13	13	-2	11	8	13	12	-2	5	9	17	-16	1	0	10	19	-18

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-6	1	10	19	18	1	1	10	12	-12	-4	3	10	6	-7	-3	4	10	9	8	-5	6	10	6	-7
-5	1	10	7	7	-6	2	10	9	10	-2	3	10	9	11	-2	4	10	8	7	-2	6	10	7	8
-3	1	10	7	-7	-4	2	10	8	-9	-1	3	10	13	14	0	4	10	7	-8	-1	6	10	6	6
-2	1	10	8	-10	-3	2	10	16	-16	0	3	10	9	9	-5	5	10	5	6	-2	7	10	6	-7
-1	1	10	14	-15	-2	2	10	15	-15	1	3	10	5	4	-2	5	10	4	-4	-2	2	11	8	8
0	1	10	19	-19	-5	3	10	10	-10	-4	4	10	12	12	0	5	10	12	11	-2	3	11	9	9

NOVA

APPENDIX NINE

COMPOUND (I)

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	63	-64	4	9	0	4	-3	4	19	0	12	13	3	3	1	16	-16	8	7	1	4	3
4	0	0	38	35	6	9	0	11	-10	6	19	0	3	3	4	3	1	39	38	0	8	1	21	21
6	0	0	23	24	0	10	0	49	48	0	20	0	3	-1	5	3	1	3	-3	1	8	1	34	-33
8	0	0	6	-7	2	10	0	38	-37	2	20	0	38	39	6	3	1	5	5	2	8	1	5	4
2	1	0	123	-142	4	10	0	41	-42	4	20	0	5	5	0	4	1	34	35	3	8	1	19	18
4	1	0	5	-5	6	10	0	6	-6	2	21	0	20	19	1	4	1	11	11	4	8	1	9	-9
6	1	0	24	-23	2	11	0	10	11	4	21	0	22	-22	2	4	1	9	9	5	8	1	4	5
8	1	0	3	-4	4	11	0	11	-11	0	22	0	7	-8	3	4	1	8	-8	7	8	1	5	3
2	2	0	127	-147	6	11	0	3	0	2	22	0	3	2	4	4	1	11	10	1	9	1	2	-2
4	2	0	5	-5	0	12	0	60	59	4	22	0	12	-12	5	4	1	14	14	2	9	1	41	-43
6	2	0	5	5	2	12	0	36	35	2	23	0	12	12	6	4	1	2	-3	3	9	1	8	8
2	3	0	7	-6	4	12	0	3	-2	4	23	0	15	-15	1	5	1	2	-1	4	9	1	8	8
4	3	0	40	39	6	12	0	24	23	2	24	0	6	-6	2	5	1	33	-33	5	9	1	14	14
6	3	0	12	-12	2	13	0	4	5	4	24	0	7	-7	3	5	1	3	-2	6	9	1	9	-9
0	4	0	73	80	4	13	0	21	-22	2	25	0	4	4	4	5	1	21	20	7	9	1	11	-11
2	4	0	29	-31	6	13	0	13	-13	0	26	0	5	-5	5	5	1	15	15	0	10	1	40	41
4	4	0	18	-17	0	14	0	3	-2	2	26	0	4	3	6	5	1	2	-3	1	10	1	9	-8
2	5	0	33	-33	2	14	0	19	-19	1	1	1	60	63	7	5	1	16	-16	2	10	1	7	-8
4	5	0	5	-4	4	14	0	20	-20	2	1	1	74	-77	8	5	1	8	8	3	10	1	39	38
6	5	0	8	-8	6	14	0	7	7	3	1	1	36	-36	0	6	1	47	47	4	10	1	14	-14
8	5	0	5	-4	2	15	0	17	16	4	1	1	14	15	1	6	1	31	30	5	10	1	24	-24
0	6	0	19	20	4	15	0	2	1	5	1	1	30	29	2	6	1	2	-3	6	10	1	3	2
2	6	0	49	-51	6	15	0	19	18	6	1	1	7	-6	3	6	1	10	-9	7	10	1	6	7
4	6	0	14	-14	0	16	0	18	17	7	1	1	8	-8	5	6	1	2	-3	1	11	1	32	30
8	6	0	3	-3	2	16	0	9	-10	8	1	1	5	-5	6	6	1	8	8	2	11	1	45	-46
2	7	0	2	2	4	16	0	3	4	1	2	1	75	-81	7	6	1	5	-4	3	11	1	31	-29
4	7	0	26	27	6	16	0	9	-9	2	2	1	85	-86	8	6	1	4	4	4	11	1	6	6
8	7	0	10	10	2	17	0	4	4	3	2	1	5	5	1	7	1	9	9	5	11	1	2	-2
0	8	0	32	32	4	17	0	10	-9	4	2	1	19	-17	2	7	1	35	-34	6	11	1	4	-4
2	8	0	11	10	6	17	0	4	-4	5	2	1	4	-4	3	7	1	28	-28	0	12	1	16	16
4	8	0	27	26	0	18	0	12	-12	6	2	1	8	-8	4	7	1	31	30	1	12	1	59	-56
6	8	0	7	7	2	18	0	15	-15	7	2	1	9	-9	5	7	1	19	19	2	12	1	4	4
8	8	0	8	7	4	18	0	6	-5	1	3	1	35	-35	6	7	1	4	3	3	12	1	5	4
2	9	0	61	60	2	19	0	15	15	2	3	1	52	-52	7	7	1	9	-10	4	12	1	5	5

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
5	12	1	6	-5	3	18	1	4	-5	1	27	1	3	-2	2	4	2	44	43	6	8	2	14	-15
6	12	1	14	13	4	18	1	5	5	0	0	2	74	79	3	4	2	16	16	7	8	2	7	-6
7	12	1	4	-4	5	18	1	3	3	1	0	2	119	-138	4	4	2	23	22	1	9	2	8	7
1	13	1	3	-3	6	18	1	4	4	2	0	2	125	-136	5	4	2	12	-11	3	9	2	34	-33
2	13	1	12	-12	1	19	1	20	-20	3	0	2	33	-36	6	4	2	3	-3	4	9	2	6	6
3	13	1	14	15	2	19	1	3	4	4	0	2	7	6	7	4	2	2	-2	5	9	2	12	-11
4	13	1	14	14	3	19	1	20	20	5	0	2	22	-22	1	5	2	38	-37	6	9	2	9	-8
6	13	1	3	2	5	19	1	8	-8	6	0	2	7	-7	2	5	2	20	-19	7	9	2	8	-8
7	13	1	16	15	0	20	1	37	38	7	0	2	14	-14	3	5	2	15	13	0	10	2	7	-7
0	14	1	13	-11	1	20	1	29	29	8	0	2	8	-8	4	5	2	13	13	1	10	2	15	-14
1	14	1	4	-5	2	20	1	6	-5	1	1	2	121	-137	5	5	2	10	-11	2	10	2	22	22
2	14	1	22	-21	3	20	1	3	2	2	1	2	58	-59	6	5	2	3	3	3	10	2	16	-15
3	14	1	3	-2	4	20	1	2	-1	3	1	2	29	-30	7	5	2	2	2	4	10	2	8	9
4	14	1	19	-19	1	21	1	16	15	4	1	2	34	35	0	6	2	38	-37	5	10	2	2	2
5	14	1	10	9	2	21	1	14	-14	5	1	2	7	-7	1	6	2	20	19	1	11	2	20	20
6	14	1	8	-7	3	21	1	9	10	6	1	2	2	-2	2	6	2	8	8	2	11	2	18	-18
7	14	1	7	-8	4	21	1	6	-6	7	1	2	3	-3	3	6	2	21	21	3	11	2	9	9
1	15	1	18	-17	5	21	1	6	-6	8	1	2	4	4	4	6	2	19	20	4	11	2	8	8
2	15	1	4	-4	0	22	1	5	-6	0	2	2	28	-28	6	6	2	10	-11	5	11	2	22	22
3	15	1	8	8	1	22	1	19	20	1	2	2	39	-40	7	6	2	9	9	6	11	2	3	-3
4	15	1	7	7	2	22	1	22	-23	2	2	2	10	-11	8	6	2	10	-9	7	11	2	3	2
5	15	1	16	-16	3	22	1	21	-22	3	2	2	63	63	1	7	2	30	30	0	12	2	56	-54
6	15	1	10	10	4	22	1	8	-8	4	2	2	37	38	2	7	2	46	-45	1	12	2	17	-17
0	16	1	25	24	1	23	1	7	-7	5	2	2	7	7	3	7	2	20	20	2	12	2	6	5
1	16	1	10	11	2	23	1	3	2	6	2	2	5	-5	4	7	2	9	9	4	12	2	10	-10
3	16	1	6	-6	3	23	1	12	-12	1	3	2	17	17	5	7	2	9	9	5	12	2	9	-9
5	16	1	4	5	4	23	1	6	7	2	3	2	22	-22	6	7	2	12	-12	6	12	2	5	4
2	17	1	3	-3	0	24	1	20	19	3	3	2	28	27	7	7	2	8	8	7	12	2	9	-8
3	17	1	6	6	1	24	1	11	10	4	3	2	31	31	0	8	2	74	-74	1	13	2	24	-23
4	17	1	3	-2	1	25	1	7	7	5	3	2	8	-8	1	8	2	26	25	2	13	2	13	-13
6	17	1	2	3	2	25	1	4	-4	6	3	2	7	-7	2	8	2	4	4	3	13	2	27	-27
0	18	1	27	27	3	25	1	3	-4	8	3	2	3	4	3	8	2	4	3	4	13	2	18	19
1	18	1	7	-7	0	26	1	3	3	0	4	2	29	-29	4	8	2	21	21	5	13	2	3	-1
2	18	1	6	-6	2	26	1	4	-4	1	4	2	52	-53	5	8	2	7	-6	6	13	2	5	4

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
7	13	2	3	-3	2	19	2	17	-16	0	2	3	32	31	6	6	3	3	2	7	11	3	12	12
0	14	2	28	-27	3	19	2	7	7	1	2	3	49	-49	7	6	3	6	-6	0	12	3	46	-46
1	14	2	13	-13	4	19	2	7	7	2	2	3	11	-12	1	7	3	7	6	1	12	3	29	-28
2	14	2	16	17	5	19	2	7	7	3	2	3	39	39	2	7	3	21	22	2	12	3	5	5
3	14	2	17	17	0	20	2	40	-41	4	2	3	25	25	3	7	3	3	-4	3	12	3	23	23
4	14	2	4	-4	1	20	2	4	2	5	2	3	12	12	4	7	3	4	4	4	12	3	6	4
5	14	2	17	16	2	20	2	11	10	6	2	3	2	-1	5	7	3	6	-6	5	12	3	7	6
6	14	2	7	-7	3	20	2	9	-9	7	2	3	3	2	6	7	3	10	-10	1	13	3	16	-16
7	14	2	6	5	4	20	2	7	-7	1	3	3	30	29	7	7	3	6	-6	2	13	3	15	15
1	15	2	7	6	5	20	2	10	-9	2	3	3	21	21	0	8	3	13	-14	4	13	3	4	3
2	15	2	15	-16	1	21	2	8	-8	3	3	3	40	41	1	8	3	39	-38	5	13	3	22	-22
3	15	2	11	12	2	21	2	5	6	4	3	3	18	17	2	8	3	23	23	0	14	3	22	-21
4	15	2	2	0	3	21	2	14	-14	5	3	3	6	-5	4	8	3	6	6	1	14	3	9	-8
5	15	2	3	2	4	21	2	5	-5	6	3	3	11	-11	5	8	3	13	-13	2	14	3	21	20
6	15	2	10	9	0	22	2	3	-3	7	3	3	8	8	6	8	3	7	7	3	14	3	7	6
1	16	2	4	3	1	22	2	4	4	0	4	3	16	-16	7	8	3	9	-9	4	14	3	5	4
2	16	2	10	9	2	22	2	8	7	1	4	3	8	-8	1	9	3	19	-19	5	14	3	9	9
3	16	2	5	-4	3	22	2	4	-4	2	4	3	12	12	2	9	3	25	25	6	14	3	8	-8
4	16	2	16	16	4	22	2	3	-3	4	4	3	19	18	3	9	3	6	6	1	15	3	12	12
5	16	2	11	-11	3	23	2	7	-7	5	4	3	3	-3	4	9	3	3	3	2	15	3	13	12
6	16	2	3	-3	0	24	2	5	6	6	4	3	17	-17	5	9	3	4	-4	3	15	3	21	22
1	17	2	8	-8	1	24	2	5	5	7	4	3	6	6	6	9	3	11	-11	4	15	3	17	-17
2	17	2	14	-14	3	24	2	4	-4	1	5	3	8	7	7	9	3	4	4	5	15	3	3	-1
3	17	2	6	-6	1	25	2	2	2	2	5	3	30	30	1	10	3	21	-20	6	15	3	4	4
4	17	2	5	4	2	25	2	5	-5	3	5	3	6	5	3	10	3	22	21	0	16	3	12	-12
5	17	2	7	7	0	26	2	4	-3	4	5	3	17	-16	4	10	3	11	-10	1	16	3	4	-4
6	17	2	3	-4	1	26	2	4	4	6	5	3	25	-25	5	10	3	15	-14	2	16	3	6	5
0	18	2	12	-13	1	1	3	23	-22	7	5	3	4	4	6	10	3	2	-1	3	16	3	3	-2
1	18	2	9	9	2	1	3	10	8	0	6	3	26	-25	7	10	3	5	4	4	16	3	16	15
2	18	2	7	6	3	1	3	44	-42	1	6	3	12	-12	1	11	3	6	5	5	16	3	6	-6
3	18	2	10	10	4	1	3	2	1	2	6	3	14	-14	2	11	3	11	-10	1	17	3	5	5
4	18	2	3	4	5	1	3	2	1	3	6	3	7	6	3	11	3	4	-4	2	17	3	17	17
5	18	2	3	3	6	1	3	7	-6	4	6	3	6	-6	4	11	3	3	1	3	17	3	3	0
1	19	2	22	22	7	1	3	3	-2	5	6	3	4	3	6	11	3	2	-2	4	17	3	16	-18

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
6	17	3	3	3	4	0	4	17	-17	0	6	4	65	66	7	10	4	2	3	3	17	4	4	-3
0	18	3	9	-9	5	0	4	22	22	1	6	4	15	15	1	11	4	15	-14	4	17	4	11	11
1	18	3	19	-18	6	0	4	10	11	2	6	4	4	5	3	11	4	8	-9	5	17	4	5	-5
2	18	3	19	-20	7	0	4	3	4	3	6	4	14	-15	4	11	4	9	9	0	18	4	14	14
3	18	3	7	7	1	1	4	17	17	4	6	4	4	-3	5	11	4	4	-3	1	18	4	9	10
4	18	3	9	9	2	1	4	12	12	5	6	4	17	-19	6	11	4	15	-15	2	18	4	12	11
5	18	3	3	-3	3	1	4	35	36	6	6	4	15	16	0	12	4	8	-8	4	18	4	4	-3
1	19	3	7	-6	4	1	4	34	34	7	6	4	3	-2	1	12	4	2	-2	1	19	4	8	-8
2	19	3	21	22	5	1	4	14	14	1	7	4	25	-24	3	12	4	8	8	2	19	4	6	-6
3	19	3	9	9	0	2	4	56	56	2	7	4	5	-4	4	12	4	22	-23	3	19	4	8	-9
4	19	3	5	-5	1	2	4	33	-31	4	7	4	11	11	5	12	4	9	9	5	19	4	4	-3
5	19	3	10	-10	2	2	4	32	32	5	7	4	10	-10	6	12	4	5	5	0	20	4	19	-19
0	20	3	31	32	3	2	4	19	19	6	7	4	11	-12	1	13	4	13	13	1	20	4	6	6
1	20	3	3	-1	4	2	4	10	-9	7	7	4	14	15	3	13	4	8	9	2	20	4	3	3
2	20	3	7	-7	6	2	4	3	1	0	8	4	11	11	4	13	4	20	20	3	20	4	3	3
4	20	3	5	5	7	2	4	4	-4	1	8	4	33	31	6	13	4	5	-4	4	20	4	6	-6
5	20	3	6	-7	1	3	4	8	8	2	8	4	13	-13	0	14	4	3	1	1	21	4	11	11
1	21	3	16	-17	2	3	4	14	-13	4	8	4	15	-15	1	14	4	13	-12	2	21	4	10	11
2	21	3	8	-7	3	3	4	27	27	5	8	4	8	8	2	14	4	30	31	3	21	4	3	2
3	21	3	5	-4	4	3	4	4	-3	6	8	4	10	-10	3	14	4	10	-10	0	22	4	6	6
4	21	3	4	-4	5	3	4	9	-9	7	8	4	3	3	4	14	4	3	-1	3	22	4	3	-4
0	22	3	5	-4	6	3	4	10	-10	1	9	4	22	21	1	15	4	3	-3	1	23	4	4	4
1	22	3	12	13	0	4	4	24	-24	2	9	4	7	-6	2	15	4	6	5	2	23	4	3	3
2	22	3	8	-9	1	4	4	28	-27	4	9	4	4	4	3	15	4	22	22	0	24	4	6	7
4	22	3	7	7	2	4	4	6	-6	5	9	4	3	-1	4	15	4	11	-12	2	24	4	5	-5
1	23	3	13	13	3	4	4	5	5	6	9	4	9	-10	6	15	4	9	-9	1	1	5	14	13
2	23	3	5	5	4	4	4	29	-30	7	9	4	9	9	0	16	4	7	-7	2	1	5	23	-22
0	24	3	7	8	5	4	4	7	-8	0	10	4	4	4	1	16	4	11	11	3	1	5	7	7
2	25	3	7	-7	6	4	4	4	3	1	10	4	3	2	2	16	4	12	-12	4	1	5	12	11
0	26	3	8	-8	1	5	4	24	-23	2	10	4	4	5	3	16	4	8	-7	5	1	5	14	15
0	0	4	4	-3	2	5	4	17	18	3	10	4	13	-13	4	16	4	8	-8	6	1	5	14	14
1	0	4	44	-43	3	5	4	22	22	4	10	4	4	-4	5	16	4	6	-7	7	1	5	3	-3
2	0	4	48	-48	5	5	4	22	-23	5	10	4	12	12	1	17	4	10	-10	0	2	5	17	16
3	0	4	10	10	6	5	4	4	4	6	10	4	7	7	2	17	4	10	10	1	2	5	29	-30

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	2	5	7	6	3	7	5	13	14	3	13	5	17	17	0	22	5	12	12	1	6	6	17	16
3	2	5	16	16	4	7	5	8	-8	5	13	5	5	-5	1	22	5	4	4	2	6	6	22	-24
4	2	5	13	13	5	7	5	2	3	6	13	5	7	6	2	22	5	6	6	4	6	6	4	3
5	2	5	7	-7	6	7	5	3	2	0	14	5	10	-10	0	0	6	10	-11	5	6	6	7	-6
6	2	5	5	-5	0	8	5	21	-21	2	14	5	25	26	1	0	6	11	11	6	6	6	8	8
7	2	5	3	-4	1	8	5	14	-13	3	14	5	11	-11	2	0	6	18	19	1	7	6	6	-6
1	3	5	18	-18	2	8	5	3	2	4	14	5	5	-4	3	0	6	2	2	2	7	6	3	-3
2	3	5	32	-32	3	8	5	9	8	5	14	5	5	-5	4	0	6	26	27	3	7	6	5	4
3	3	5	10	8	4	8	5	17	-18	1	15	5	12	-12	5	0	6	9	9	4	7	6	16	17
4	3	5	8	8	5	8	5	5	6	2	15	5	4	-2	1	1	6	8	9	5	7	6	18	-18
5	3	5	18	-18	6	8	5	6	6	4	15	5	12	-12	3	1	6	2	2	6	7	6	10	10
7	3	5	6	-7	1	9	5	5	-6	0	16	5	15	-15	5	1	6	3	-3	0	8	6	16	16
0	4	5	27	-26	2	9	5	3	-2	1	16	5	6	5	6	1	6	8	-8	1	8	6	18	18
1	4	5	3	-3	3	9	5	7	8	2	16	5	3	3	0	2	6	29	-28	2	8	6	4	3
2	4	5	11	10	4	9	5	4	4	3	16	5	5	5	1	2	6	8	-8	3	8	6	12	-12
3	4	5	2	-2	5	9	5	12	-13	4	16	5	7	-7	2	2	6	9	9	4	8	6	15	14
4	4	5	3	3	6	9	5	10	10	1	17	5	6	-6	3	2	6	7	-6	6	8	6	6	-7
5	4	5	20	-20	0	10	5	10	10	2	17	5	8	9	4	2	6	3	3	1	9	6	4	-4
6	4	5	6	-6	1	10	5	10	-9	3	17	5	13	-14	5	2	6	6	-6	2	9	6	16	-16
7	4	5	3	2	2	10	5	12	12	4	17	5	12	-12	6	2	6	9	-9	3	9	6	4	4
1	5	5	17	-16	4	10	5	12	-12	0	18	5	9	9	1	3	6	27	-26	4	9	6	3	3
2	5	5	8	-8	6	10	5	5	4	1	18	5	11	-12	3	3	6	3	-3	5	9	6	5	-5
3	5	5	14	-13	1	11	5	10	-10	2	18	5	5	-5	4	3	6	11	-12	6	9	6	4	4
5	5	5	3	0	2	11	5	14	-14	3	18	5	13	14	5	3	6	8	-7	0	10	6	16	-15
6	5	5	4	-4	3	11	5	7	7	1	19	5	11	11	0	4	6	29	-29	1	10	6	12	12
7	5	5	11	11	4	11	5	8	7	2	19	5	7	6	2	4	6	3	2	2	10	6	4	-4
0	6	5	15	-15	6	11	5	10	10	3	19	5	9	9	3	4	6	13	14	3	10	6	7	-6
1	6	5	10	-10	0	12	5	10	-11	4	19	5	6	-7	4	4	6	3	2	4	10	6	11	11
2	6	5	17	17	1	12	5	3	-3	0	20	5	10	10	1	5	6	7	7	6	10	6	4	-4
3	6	5	12	11	2	12	5	15	15	1	20	5	11	-12	2	5	6	8	8	2	11	6	5	-5
4	6	5	14	-15	3	12	5	5	5	2	20	5	11	-11	3	5	6	31	31	3	11	6	6	6
5	6	5	12	11	4	12	5	10	-10	3	20	5	3	-2	4	5	6	17	-17	4	11	6	7	7
1	7	5	11	10	1	13	5	7	7	1	21	5	5	-5	5	5	6	5	-3	5	11	6	5	-5
2	7	5	10	-11	2	13	5	9	-9	3	21	5	8	8	6	5	6	3	2	1	12	6	5	5

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	12	6	11	12	2	2	7	9	-10	4	9	7	12	-12	4	0	8	4	-4	0	10	8	11	10
4	12	6	6	6	3	2	7	8	8	0	10	7	12	-12	1	1	8	4	3	2	10	8	7	-7
1	13	6	2	1	4	2	7	4	-5	1	10	7	6	6	3	1	8	7	-7	3	10	8	3	3
2	13	6	17	-17	5	2	7	5	-5	2	10	7	2	2	4	1	8	3	-2	2	11	8	4	5
3	13	6	13	13	6	2	7	7	-7	3	10	7	9	-9	0	2	8	3	-2	0	12	8	5	6
5	13	6	5	-6	1	3	7	4	4	4	10	7	5	5	1	2	8	14	-14	1	12	8	6	-5
0	14	6	31	-31	3	3	7	3	2	5	10	7	3	3	2	2	8	4	-4	2	12	8	6	-5
1	14	6	8	7	5	3	7	5	4	2	11	7	10	9	4	2	8	4	-4	1	13	8	7	-6
2	14	6	8	8	6	3	7	9	-9	4	11	7	5	-5	2	3	8	17	16	2	13	8	6	5
3	14	6	8	-8	0	4	7	28	-28	5	11	7	5	5	4	3	8	3	-3	0	14	8	4	-5
4	14	6	4	4	1	4	7	13	12	0	12	7	7	-8	5	3	8	4	4	1	14	8	4	4
1	15	6	20	-21	2	4	7	7	7	1	12	7	9	9	0	4	8	26	26	2	14	8	3	-2
2	15	6	6	6	3	4	7	3	3	2	12	7	6	7	1	4	8	19	-20	1	15	8	3	1
3	15	6	4	4	5	4	7	10	-10	3	12	7	5	-5	2	4	8	8	8	2	15	8	15	16
4	15	6	8	-8	1	5	7	13	14	4	13	7	6	-6	3	4	8	9	10	0	16	8	22	22
0	16	6	8	-6	2	5	7	17	17	0	14	7	28	-28	1	5	8	9	-8	1	16	8	9	9
1	16	6	21	22	3	5	7	18	-19	1	14	7	15	15	2	5	8	13	14	1	1	9	7	-7
2	16	6	7	-7	4	5	7	10	10	3	14	7	6	-7	4	5	8	11	-11	2	1	9	8	-8
4	16	6	4	-4	5	5	7	5	5	4	14	7	3	-4	0	6	8	28	28	3	1	9	5	4
1	17	6	8	9	0	6	7	14	-14	1	15	7	4	0	1	6	8	9	-10	0	2	9	14	14
3	17	6	11	11	2	6	7	29	29	2	15	7	12	11	2	6	8	16	-16	2	2	9	3	3
4	17	6	3	3	3	6	7	6	-5	3	15	7	6	-7	3	6	8	11	11	1	3	9	7	-7
1	18	6	11	11	4	6	7	3	-4	0	16	7	11	-11	4	6	8	11	-11	2	3	9	16	-16
3	18	6	10	-10	5	6	7	3	3	1	16	7	14	13	1	7	8	9	9	3	3	9	7	8
2	19	6	5	-5	2	7	7	14	14	2	16	7	8	8	2	7	8	5	-5	0	4	9	4	-4
1	20	6	4	4	3	7	7	16	-16	1	17	7	11	11	3	7	8	10	9	1	4	9	14	14
2	20	6	10	10	4	7	7	9	-9	2	17	7	10	10	4	7	8	4	-4	2	4	9	4	3
2	21	6	3	-3	0	8	7	12	-12	3	17	7	9	-9	0	8	8	14	14	3	4	9	4	4
2	1	7	6	7	2	8	7	9	9	0	18	7	9	9	1	8	8	8	-8	1	5	9	10	10
3	1	7	10	-10	4	8	7	5	-5	1	18	7	3	-3	2	8	8	10	-10	2	5	9	16	-15
5	1	7	8	8	5	8	7	7	7	2	18	7	6	6	3	8	8	6	-7	0	6	9	15	-15
6	1	7	6	-7	1	9	7	9	9	0	0	8	5	5	1	9	8	4	-5	2	6	9	6	5
0	2	7	12	-11	2	9	7	7	7	1	0	8	3	2	3	9	8	4	4	3	6	9	7	-7
1	2	7	6	-7	3	9	7	3	4	2	0	8	9	-9	4	9	8	3	-4	2	7	9	7	-8

OBSERVED AND CALCULATED STRUCTURE FACTORS .

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	8	9	4	3	2	9	9	5	-6	2	10	9	6	-6	0	0	10	7	-7	0	2	10	19	-18
2	8	9	4	5	0	10	9	3	2	0	12	9	10	9	1	0	10	9	8	1	3	10	9	9
1	9	9	7	-7	1	10	9	6	5	1	12	9	3	1	1	1	10	4	4	0	4	10	16	-15

1846:>

COMPOUND (II)
OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	0	0	52	-45	11	1	0	40	34	19	2	0	32	29	8	4	0	3	3	-15	1	1	8	6
5	0	0	27	20	12	1	0	14	10	20	2	0	15	13	9	4	0	6	6	-14	1	1	24	26
6	0	0	27	-23	13	1	0	19	-17	21	2	0	23	-22	11	4	0	12	-11	-13	1	1	18	-16
7	0	0	48	40	14	1	0	25	22	22	2	0	14	-14	14	4	0	17	-16	-12	1	1	17	17
8	0	0	26	-21	15	1	0	13	-11	23	2	0	4	-4	15	4	0	13	12	-11	1	1	11	11
9	0	0	29	-24	16	1	0	14	-13	25	2	0	4	-4	16	4	0	4	4	-10	1	1	21	-22
10	0	0	13	-10	17	1	0	12	-11	1	3	0	6	6	17	4	0	12	-11	-9	1	1	38	37
11	0	0	30	25	18	1	0	11	10	4	3	0	19	17	18	4	0	6	5	-8	1	1	8	7
12	0	0	74	-66	19	1	0	25	23	5	3	0	14	-13	19	4	0	8	7	-7	1	1	13	13
13	0	0	67	-57	20	1	0	28	-27	6	3	0	7	-7	21	4	0	9	-8	-6	1	1	3	-4
14	0	0	34	29	21	1	0	4	4	7	3	0	7	6	1	5	0	8	-8	-5	1	1	25	26
15	0	0	26	21	22	1	0	4	-4	8	3	0	3	-2	2	5	0	4	2	-4	1	1	10	-11
16	0	0	15	13	23	1	0	19	-20	9	3	0	24	-22	3	5	0	19	17	-3	1	1	6	8
17	0	0	11	10	24	1	0	8	7	10	3	0	7	-7	4	5	0	8	6	-1	1	1	66	-92
18	0	0	7	6	25	1	0	4	-2	11	3	0	5	-4	5	5	0	15	-14	0	1	1	46	55
19	0	0	24	-24	27	1	0	7	-7	12	3	0	3	-2	6	5	0	17	15	1	1	1	12	-12
20	0	0	11	11	28	1	0	6	-7	13	3	0	6	-5	7	5	0	3	-1	2	1	1	49	47
21	0	0	5	-6	0	2	0	20	-20	14	3	0	12	-10	8	5	0	9	-9	3	1	1	4	-4
22	0	0	7	-7	1	2	0	7	7	15	3	0	14	13	9	5	0	12	12	4	1	1	17	14
23	0	0	6	6	2	2	0	5	-4	16	3	0	17	-16	10	5	0	6	5	5	1	1	29	25
24	0	0	14	-15	3	2	0	7	6	17	3	0	7	-4	11	5	0	4	-4	6	1	1	25	21
25	0	0	9	-9	4	2	0	32	28	18	3	0	19	19	12	5	0	5	-5	7	1	1	16	-14
26	0	0	10	-11	5	2	0	47	-40	21	3	0	8	8	-27	1	1	9	9	8	1	1	4	5
27	0	0	8	7	6	2	0	37	-32	22	3	0	16	16	-26	1	1	10	11	9	1	1	22	20
28	0	0	9	10	7	2	0	45	-39	23	3	0	4	3	-25	1	1	5	3	10	1	1	17	-15
29	0	0	6	6	8	2	0	11	-9	24	3	0	5	-4	-24	1	1	9	-8	11	1	1	33	30
1	1	0	26	28	9	2	0	25	21	0	4	0	7	7	-23	1	1	24	26	12	1	1	5	-5
3	1	0	17	17	10	2	0	28	-24	1	4	0	15	13	-22	1	1	7	-4	13	1	1	22	-20
4	1	0	50	45	11	2	0	16	14	2	4	0	15	14	-21	1	1	18	-19	14	1	1	27	-27
5	1	0	30	26	12	2	0	8	7	3	4	0	10	-9	-20	1	1	6	-5	15	1	1	17	-17
6	1	0	20	-16	14	2	0	15	13	4	4	0	3	-4	-19	1	1	9	-9	16	1	1	3	3
7	1	0	28	24	15	2	0	9	7	5	4	0	14	14	-18	1	1	8	-6	17	1	1	5	-6
8	1	0	43	34	17	2	0	6	6	6	4	0	9	-8	-17	1	1	19	-19	18	1	1	12	-12
10	1	0	8	-8	18	2	0	28	26	7	4	0	15	-14	-16	1	1	7	-6	19	1	1	6	5

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
20	1	1	25	27	5	2	1	31	-29	0	3	1	10	-10	-4	4	1	9	-9	10	5	1	7	6
21	1	1	8	-9	6	2	1	3	-3	1	3	1	7	6	-3	4	1	7	6	11	5	1	7	6
22	1	1	4	4	7	2	1	32	29	2	3	1	27	-26	-2	4	1	3	3	13	5	1	6	-5
23	1	1	16	18	8	2	1	4	-3	3	3	1	4	3	-1	4	1	7	8	-29	0	2	9	-10
24	1	1	9	10	9	2	1	16	-13	4	3	1	38	37	1	4	1	14	-16	-27	0	2	10	-11
25	1	1	7	-7	10	2	1	21	19	5	3	1	13	13	2	4	1	16	-16	-25	0	2	14	-15
26	1	1	5	-5	11	2	1	3	-1	6	3	1	13	11	3	4	1	6	6	-24	0	2	15	-16
28	1	1	4	-5	12	2	1	7	-7	7	3	1	14	-12	4	4	1	10	9	-23	0	2	28	31
-26	2	1	6	5	14	2	1	12	12	8	3	1	3	4	5	4	1	5	-6	-22	0	2	12	-12
-25	2	1	5	3	16	2	1	9	-9	9	3	1	4	-2	6	4	1	7	7	-21	0	2	20	20
-24	2	1	12	-12	17	2	1	5	-4	10	3	1	3	-3	10	4	1	6	6	-20	0	2	8	9
-23	2	1	5	2	18	2	1	7	6	11	3	1	4	3	11	4	1	4	-4	-19	0	2	14	-14
-21	2	1	4	5	19	2	1	3	-2	13	3	1	6	-6	12	4	1	6	6	-18	0	2	19	18
-20	2	1	15	16	20	2	1	16	-16	15	3	1	20	19	14	4	1	3	-3	-17	0	2	4	3
-19	2	1	7	5	21	2	1	16	16	16	3	1	27	-26	15	4	1	15	15	-16	0	2	13	-15
-18	2	1	6	-6	22	2	1	7	-6	17	3	1	27	-26	16	4	1	15	-14	-15	0	2	17	-19
-17	2	1	21	-22	24	2	1	12	13	18	3	1	8	-7	17	4	1	5	-3	-14	0	2	23	21
-16	2	1	5	-1	25	2	1	4	-5	19	3	1	11	10	19	4	1	5	-4	-12	0	2	8	-9
-15	2	1	13	14	-24	3	1	11	10	20	3	1	23	23	-14	5	1	4	4	-11	0	2	6	-6
-14	2	1	5	-4	-23	3	1	4	3	21	3	1	10	9	-13	5	1	5	7	-10	0	2	18	19
-13	2	1	6	-5	-22	3	1	4	3	23	3	1	8	-8	-12	5	1	6	-5	-9	0	2	5	-5
-12	2	1	5	4	-21	3	1	10	-10	-21	4	1	7	-7	-11	5	1	4	-5	-8	0	2	23	-23
-11	2	1	6	7	-20	3	1	19	-20	-20	4	1	7	-7	-10	5	1	5	4	-7	0	2	37	37
-10	2	1	16	-15	-19	3	1	6	-4	-19	4	1	8	8	-5	5	1	10	10	-6	0	2	23	24
-8	2	1	3	3	-18	3	1	9	8	-18	4	1	10	-10	-3	5	1	4	-3	-5	0	2	8	-8
-7	2	1	9	-9	-16	3	1	5	-5	-17	4	1	9	10	-2	5	1	18	-15	-4	0	2	24	28
-5	2	1	19	22	-14	3	1	7	8	-16	4	1	3	2	-1	5	1	17	-14	-2	0	2	43	-53
-4	2	1	28	34	-9	3	1	10	10	-15	4	1	10	-11	0	5	1	6	4	-1	0	2	75	-93
-3	2	1	17	-19	-8	3	1	19	20	-13	4	1	6	-8	1	5	1	8	8	0	0	2	8	9
-2	2	1	21	-25	-7	3	1	20	21	-11	4	1	4	-3	2	5	1	13	11	1	0	2	44	-46
-1	2	1	7	7	-6	3	1	9	-9	-9	4	1	5	-5	3	5	1	4	-3	2	0	2	19	-20
0	2	1	5	-6	-5	3	1	22	-24	-8	4	1	16	18	4	5	1	4	-2	3	0	2	66	-61
2	2	1	13	-15	-3	3	1	6	-6	-6	4	1	7	-9	6	5	1	4	-2	4	0	2	13	13
3	2	1	12	13	-1	3	1	4	-4	-5	4	1	14	15	7	5	1	9	6	5	0	2	39	-34

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
6	0	2	43	-36	-13	1	2	8	9	21	1	2	6	4	9	2	2	4	3	-2	3	2	3	-3
7	0	2	24	-21	-12	1	2	11	11	22	1	2	5	-7	11	2	2	3	4	-1	3	2	6	6
8	0	2	26	23	-11	1	2	13	-13	23	1	2	5	5	12	2	2	19	18	0	3	2	7	8
9	0	2	59	-54	-10	1	2	7	5	26	1	2	8	10	13	2	2	3	-2	1	3	2	8	8
10	0	2	50	-46	-9	1	2	51	48	-23	2	2	9	8	14	2	2	3	1	2	3	2	11	-11
11	0	2	86	80	-8	1	2	24	-21	-22	2	2	18	20	15	2	2	32	31	3	3	2	13	-12
12	0	2	16	14	-7	1	2	29	-28	-21	2	2	9	6	16	2	2	5	4	4	3	2	7	7
13	0	2	26	25	-6	1	2	29	-27	-19	2	2	6	7	17	2	2	4	2	6	3	2	16	-15
14	0	2	13	12	-5	1	2	13	11	-18	2	2	3	-1	18	2	2	41	-42	7	3	2	6	6
15	0	2	4	-3	-4	1	2	87	86	-17	2	2	3	3	19	2	2	23	-23	9	3	2	4	4
16	0	2	5	3	-3	1	2	13	15	-16	2	2	15	-16	20	2	2	4	4	10	3	2	5	5
17	0	2	4	-3	-2	1	2	72	83	-15	2	2	10	-7	21	2	2	10	-10	11	3	2	11	-11
18	0	2	10	10	-1	1	2	48	-43	-14	2	2	6	5	23	2	2	5	-5	12	3	2	15	16
20	0	2	3	-4	0	1	2	72	-59	-13	2	2	26	-27	-24	3	2	5	-2	13	3	2	14	-14
21	0	2	4	-5	1	1	2	84	68	-12	2	2	10	7	-23	3	2	7	7	14	3	2	3	1
22	0	2	13	-15	2	1	2	3	-2	-11	2	2	14	-14	-22	3	2	6	5	15	3	2	33	33
23	0	2	6	-2	3	1	2	38	29	-10	2	2	30	-30	-21	3	2	3	-4	16	3	2	4	-2
24	0	2	15	17	4	1	2	17	15	-9	2	2	14	-13	-20	3	2	5	4	17	3	2	3	-2
25	0	2	9	9	5	1	2	5	5	-8	2	2	8	-9	-19	3	2	9	10	18	3	2	6	7
26	0	2	7	7	6	1	2	16	-14	-7	2	2	10	10	-18	3	2	7	-7	19	3	2	7	7
27	0	2	3	-3	7	1	2	16	-14	-5	2	2	3	5	-17	3	2	5	5	22	3	2	4	-4
-28	1	2	6	-6	8	1	2	4	3	-4	2	2	14	19	-16	3	2	12	13	-20	4	2	6	-6
-26	1	2	9	9	9	1	2	25	-22	-3	2	2	22	30	-15	3	2	5	3	-19	4	2	6	6
-25	1	2	6	6	10	1	2	39	-36	-2	2	2	4	5	-14	3	2	3	-3	-18	4	2	9	9
-24	1	2	5	-4	11	1	2	11	8	-1	2	2	2	4	-13	3	2	8	9	-17	4	2	11	-12
-23	1	2	4	-1	12	1	2	17	14	0	2	2	3	3	-12	3	2	4	5	-14	4	2	4	-3
-22	1	2	22	-21	13	1	2	10	-10	1	2	2	17	17	-11	3	2	4	-4	-13	4	2	7	9
-21	1	2	17	-18	14	1	2	15	-15	2	2	2	28	-25	-10	3	2	9	-10	-12	4	2	6	7
-20	1	2	14	14	15	1	2	9	-7	3	2	2	23	-20	-9	3	2	8	-8	-10	4	2	9	-10
-19	1	2	16	17	16	1	2	3	-2	4	2	2	10	-11	-8	3	2	7	8	-9	4	2	7	-8
-17	1	2	18	-18	17	1	2	20	-21	5	2	2	8	8	-7	3	2	18	-20	-8	4	2	15	18
-16	1	2	4	-4	18	1	2	17	17	6	2	2	34	29	-5	3	2	3	2	-7	4	2	3	-1
-15	1	2	10	-10	19	1	2	15	16	7	2	2	9	-8	-4	3	2	18	-22	-6	4	2	7	-7
-14	1	2	14	-15	20	1	2	16	-18	8	2	2	10	10	-3	3	2	4	-4	-5	4	2	14	16

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	4	2	8	9	9	5	2	5	-4	7	1	3	3	-3	-5	2	3	21	-27	-16	3	3	7	-4
-3	4	2	3	3	12	5	2	8	-7	8	1	3	17	16	-4	2	3	5	5	-11	3	3	7	7
-2	4	2	7	7	-26	1	3	14	15	9	1	3	16	-15	-3	2	3	7	-6	-10	3	3	3	2
0	4	2	13	-15	-25	1	3	10	-10	10	1	3	24	-23	-2	2	3	32	30	-9	3	3	8	-9
1	4	2	8	-8	-24	1	3	20	-21	11	1	3	18	-17	-1	2	3	22	-18	-8	3	3	3	4
3	4	2	5	-5	-23	1	3	4	-3	12	1	3	12	-12	0	2	3	6	5	-7	3	3	6	-7
4	4	2	15	-15	-22	1	3	7	-7	13	1	3	5	5	1	2	3	3	1	-6	3	3	6	-6
6	4	2	6	7	-21	1	3	7	8	14	1	3	10	-11	2	2	3	45	-36	-5	3	3	14	-18
7	4	2	12	-13	-20	1	3	6	-6	15	1	3	6	-5	3	2	3	24	18	-4	3	3	12	-15
8	4	2	5	-5	-18	1	3	8	8	16	1	3	17	18	4	2	3	21	19	-3	3	3	8	10
9	4	2	5	5	-17	1	3	7	8	17	1	3	23	26	5	2	3	3	-3	-2	3	3	9	11
11	4	2	7	-7	-16	1	3	6	-6	18	1	3	7	-3	6	2	3	10	-9	-1	3	3	4	-2
12	4	2	7	8	-15	1	3	6	6	20	1	3	11	14	7	2	3	22	22	1	3	3	17	16
13	4	2	7	6	-14	1	3	7	7	21	1	3	6	6	9	2	3	5	-5	3	3	3	5	4
14	4	2	5	-6	-13	1	3	12	-12	22	1	3	13	-14	10	2	3	5	-6	4	3	3	9	-8
15	4	2	9	8	-12	1	3	29	28	23	1	3	5	-4	11	2	3	5	-5	5	3	3	5	5
16	4	2	9	10	-11	1	3	9	7	25	1	3	4	-7	12	2	3	9	9	7	3	3	10	-10
18	4	2	9	-10	-10	1	3	6	6	-25	2	3	6	6	13	2	3	11	-11	8	3	3	7	7
-13	5	2	8	-8	-9	1	3	8	-8	-24	2	3	5	4	14	2	3	11	13	10	3	3	9	-9
-11	5	2	5	6	-8	1	3	9	-9	-23	2	3	4	4	15	2	3	10	10	11	3	3	8	-8
-9	5	2	9	-10	-7	1	3	5	-5	-22	2	3	4	2	16	2	3	18	-20	12	3	3	14	15
-8	5	2	9	10	-6	1	3	12	-14	-21	2	3	11	-13	17	2	3	19	-19	13	3	3	23	-23
-6	5	2	8	-8	-5	1	3	38	-51	-20	2	3	10	-10	18	2	3	12	11	14	3	3	13	-12
-4	5	2	5	6	-4	1	3	20	-28	-19	2	3	5	4	19	2	3	5	3	15	3	3	9	9
-2	5	2	7	-7	-3	1	3	32	46	-18	2	3	9	9	20	2	3	4	4	16	3	3	8	8
-1	5	2	11	10	-2	1	3	5	-6	-17	2	3	5	-4	21	2	3	13	15	17	3	3	22	24
0	5	2	19	18	-1	1	3	18	22	-13	2	3	4	-2	22	2	3	10	-10	18	3	3	8	-5
1	5	2	3	2	0	1	3	8	8	-12	2	3	8	-9	-23	3	3	7	-7	19	3	3	5	6
2	5	2	15	-15	1	1	3	24	22	-11	2	3	3	2	-22	3	3	5	3	20	3	3	4	-3
3	5	2	10	10	2	1	3	26	25	-10	2	3	6	-5	-21	3	3	5	-5	21	3	3	4	-5
4	5	2	3	-1	3	1	3	2	1	-9	2	3	10	9	-20	3	3	5	-4	-18	4	3	8	-8
5	5	2	10	-11	4	1	3	16	-14	-8	2	3	25	24	-19	3	3	3	1	-17	4	3	4	1
6	5	2	12	12	5	1	3	5	-5	-7	2	3	18	18	-18	3	3	6	6	-11	4	3	13	13
8	5	2	5	-6	6	1	3	15	13	-6	2	3	29	-32	-17	3	3	12	14	-10	4	3	4	0

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-9	4	3	9	-11	-22	0	4	20	-19	18	0	4	6	7	11	1	4	17	-17	4	2	4	8	-8
-8	4	3	9	11	-21	0	4	13	12	19	0	4	10	-12	12	1	4	6	5	5	2	4	6	-5
-7	4	3	9	-10	-20	0	4	5	5	21	0	4	11	13	13	1	4	13	-14	6	2	4	7	-7
-6	4	3	10	10	-18	0	4	5	-4	22	0	4	4	-4	14	1	4	15	15	8	2	4	10	8
-5	4	3	9	10	-17	0	4	9	9	23	0	4	3	4	15	1	4	25	27	9	2	4	21	19
-4	4	3	3	3	-14	0	4	12	-12	24	0	4	6	-7	16	1	4	15	15	10	2	4	4	-3
-3	4	3	3	-3	-13	0	4	12	-11	-26	1	4	7	-7	17	1	4	10	-10	11	2	4	10	-11
-2	4	3	26	-28	-11	0	4	16	-16	-24	1	4	9	-9	18	1	4	10	-11	12	2	4	15	15
-1	4	3	8	-8	-10	0	4	47	49	-23	1	4	8	7	19	1	4	3	0	13	2	4	15	-14
0	4	3	10	12	-9	0	4	16	16	-22	1	4	13	13	20	1	4	6	7	14	2	4	6	-3
1	4	3	12	12	-8	0	4	26	-28	-21	1	4	10	-9	21	1	4	6	6	15	2	4	24	-27
5	4	3	4	-5	-7	0	4	6	6	-16	1	4	6	5	22	1	4	4	3	16	2	4	14	-13
8	4	3	5	-5	-6	0	4	18	21	-15	1	4	10	9	23	1	4	4	6	17	2	4	13	14
9	4	3	4	4	-5	0	4	13	-14	-13	1	4	5	5	-22	2	4	7	7	18	2	4	12	-13
10	4	3	5	7	-4	0	4	25	-29	-12	1	4	18	18	-21	2	4	3	-3	19	2	4	6	6
11	4	3	6	7	-3	0	4	12	12	-11	1	4	8	-8	-20	2	4	8	-8	21	2	4	4	5
12	4	3	4	4	-2	0	4	5	-2	-10	1	4	17	-17	-19	2	4	13	-14	22	2	4	4	5
13	4	3	18	-19	-1	0	4	58	-63	-8	1	4	13	14	-18	2	4	6	-6	-22	3	4	4	4
14	4	3	15	-14	0	0	4	32	-34	-7	1	4	25	30	-17	2	4	5	5	-21	3	4	5	-4
15	4	3	5	1	1	0	4	15	15	-6	1	4	3	-2	-16	2	4	10	-10	-19	3	4	8	8
16	4	3	7	7	2	0	4	5	-6	-5	1	4	15	17	-13	2	4	6	-7	-18	3	4	3	-1
17	4	3	5	4	3	0	4	5	-7	-4	1	4	22	-26	-11	2	4	6	5	-17	3	4	8	-8
-9	5	3	4	-3	5	0	4	24	24	-3	1	4	31	-37	-10	2	4	3	-3	-16	3	4	6	4
-6	5	3	5	-4	6	0	4	9	-9	-2	1	4	37	42	-8	2	4	23	23	-15	3	4	4	-5
-5	5	3	4	-3	7	0	4	5	-5	-1	1	4	12	-13	-7	2	4	22	23	-14	3	4	5	-3
-4	5	3	3	-3	8	0	4	52	53	0	1	4	13	13	-6	2	4	36	42	-13	3	4	4	-3
-2	5	3	6	6	10	0	4	6	7	1	1	4	3	-2	-5	2	4	15	-18	-12	3	4	9	-9
1	5	3	4	3	11	0	4	22	21	2	1	4	22	-20	-4	2	4	17	-16	-11	3	4	8	7
8	5	3	4	4	12	0	4	14	13	3	1	4	13	-12	-3	2	4	3	-1	-10	3	4	15	-16
9	5	3	4	-4	13	0	4	11	11	4	1	4	15	-14	-2	2	4	9	-9	-9	3	4	5	-3
-27	0	4	6	-2	14	0	4	20	-20	6	1	4	14	-14	-1	2	4	9	9	-8	3	4	8	8
-26	0	4	31	31	15	0	4	11	-12	7	1	4	8	-8	0	2	4	17	-14	-7	3	4	11	-13
-25	0	4	7	6	16	0	4	9	-9	9	1	4	35	32	2	2	4	7	6	-5	3	4	5	6
-24	0	4	8	8	17	0	4	8	-7	10	1	4	12	-12	3	2	4	12	9	-4	3	4	7	9

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	3	4	10	11	10	4	4	7	8	7	1	5	12	-11	7	2	5	7	-7	15	3	5	7	-7
-2	3	4	5	4	12	4	4	5	3	8	1	5	6	-5	8	2	5	6	-5	16	3	5	11	11
-1	3	4	11	-10	13	4	4	7	7	9	1	5	3	-3	9	2	5	15	14	-14	4	5	4	4
0	3	4	5	-3	14	4	4	4	-5	10	1	5	6	-5	10	2	5	15	-15	-11	4	5	7	7
1	3	4	7	7	-7	5	4	4	5	11	1	5	7	-7	11	2	5	9	9	-10	4	5	4	-3
2	3	4	6	6	-5	5	4	5	-5	12	1	5	9	9	12	2	5	7	7	-6	4	5	4	-4
4	3	4	5	4	-4	5	4	9	8	13	1	5	21	21	13	2	5	24	-25	-5	4	5	12	-12
8	3	4	5	-6	-3	5	4	8	8	14	1	5	22	24	14	2	5	6	-2	-3	4	5	9	9
9	3	4	7	7	-2	5	4	6	-5	15	1	5	6	-7	15	2	5	7	7	-2	4	5	5	5
10	3	4	9	-9	-1	5	4	8	-9	16	1	5	7	-8	16	2	5	7	7	-1	4	5	3	-3
11	3	4	5	4	0	5	4	6	5	17	1	5	5	6	18	2	5	4	5	2	4	5	3	-4
12	3	4	22	22	2	5	4	9	-10	18	1	5	4	2	19	2	5	3	-5	5	4	5	4	-2
13	3	4	7	-5	-24	1	5	7	7	19	1	5	12	-12	-20	3	5	7	8	6	4	5	4	5
14	3	4	10	-11	-21	1	5	4	5	-24	2	5	7	-7	-19	3	5	7	-7	7	4	5	5	5
15	3	4	3	1	-17	1	5	3	3	-22	2	5	4	3	-11	3	5	11	11	8	4	5	5	5
17	3	4	7	-8	-15	1	5	14	13	-21	2	5	4	-4	-10	3	5	14	-12	9	4	5	6	-5
19	3	4	5	-5	-14	1	5	4	4	-15	2	5	3	-4	-9	3	5	16	-13	10	4	5	12	-12
-15	4	4	4	5	-13	1	5	4	-3	-14	2	5	6	6	-8	3	5	17	-17	11	4	5	9	-8
-14	4	4	3	-3	-12	1	5	10	-11	-11	2	5	13	13	-7	3	5	8	-7	-24	0	6	4	3
-13	4	4	9	-8	-11	1	5	13	-13	-10	2	5	6	-4	-6	3	5	34	30	-23	0	6	4	4
-12	4	4	4	-3	-10	1	5	4	2	-9	2	5	9	-10	-5	3	5	11	9	-22	0	6	4	-3
-11	4	4	9	9	-9	1	5	6	-6	-8	2	5	5	-5	-4	3	5	7	6	-21	0	6	4	-3
-9	4	4	6	-7	-8	1	5	12	-13	-7	2	5	4	4	-3	3	5	9	-8	-19	0	6	3	-3
-8	4	4	9	9	-6	1	5	13	15	-5	2	5	9	12	-2	3	5	5	-4	-18	0	6	5	-4
-7	4	4	4	4	-4	1	5	3	3	-4	2	5	12	-13	-1	3	5	8	6	-17	0	6	10	-9
-4	4	4	6	-7	-3	1	5	12	13	-3	2	5	5	3	2	3	5	3	3	-16	0	6	9	-9
-3	4	4	11	-12	-2	1	5	17	19	-2	2	5	10	10	4	3	5	7	-6	-15	0	6	4	4
-2	4	4	4	-3	0	1	5	3	-3	-1	2	5	22	-20	5	3	5	9	9	-13	0	6	21	22
1	4	4	8	-8	1	1	5	11	-11	0	2	5	17	13	6	3	5	7	-6	-12	0	6	5	3
3	4	4	11	12	2	1	5	5	-4	1	2	5	5	3	7	3	5	17	-17	-11	0	6	19	-19
4	4	4	11	-11	3	1	5	9	8	2	2	5	4	-4	8	3	5	6	-4	-10	0	6	4	3
5	4	4	4	3	4	1	5	5	-4	4	2	5	14	14	9	3	5	9	9	-7	0	6	3	-4
6	4	4	9	8	5	1	5	3	3	5	2	5	4	2	12	3	5	7	7	-6	0	6	3	3
8	4	4	5	6	6	1	5	13	-13	6	2	5	8	-7	14	3	5	7	7	-5	0	6	4	1

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	0	6	37	-38	9	1	6	19	18	-7	3	6	3	2	0	1	7	5	5	-6	3	7	5	-6
-3	0	6	5	-4	10	1	6	12	-12	-6	3	6	4	4	1	1	7	4	-3	-5	3	7	5	-5
-2	0	6	22	23	11	1	6	15	15	-5	3	6	5	5	3	1	7	6	-6	-4	3	7	8	8
-1	0	6	5	5	12	1	6	10	10	-4	3	6	12	-11	4	1	7	4	-4	-3	3	7	5	-4
0	0	6	13	13	15	1	6	13	-14	-3	3	6	4	3	7	1	7	8	-8	-1	3	7	3	2
2	0	6	10	10	16	1	6	6	6	-2	3	6	5	4	8	1	7	3	1	2	3	7	4	2
5	0	6	14	15	-20	2	6	5	6	0	3	6	5	4	9	1	7	15	15	3	3	7	9	-8
8	0	6	17	17	-14	2	6	6	5	1	3	6	6	-5	10	1	7	11	11	4	3	7	14	-13
9	0	6	21	20	-11	2	6	17	18	2	3	6	3	-3	11	1	7	14	15	5	3	7	5	4
10	0	6	17	-16	-10	2	6	10	10	3	3	6	4	-3	12	1	7	12	-12	6	3	7	6	6
11	0	6	26	-28	-9	2	6	14	15	4	3	6	4	-4	13	1	7	9	-9	7	3	7	8	8
12	0	6	26	-27	-8	2	6	19	-22	7	3	6	6	-6	14	1	7	4	5	-16	0	8	5	4
13	0	6	9	-9	-7	2	6	18	-20	8	3	6	3	4	-15	2	7	4	-3	-12	0	8	5	4
14	0	6	12	14	-6	2	6	5	2	9	3	6	8	7	-14	2	7	6	6	-11	0	8	6	-6
15	0	6	8	8	-5	2	6	6	-7	10	3	6	3	-4	-13	2	7	5	-5	-10	0	8	5	-4
16	0	6	5	-5	-4	2	6	11	12	11	3	6	8	-8	-12	2	7	5	5	-9	0	8	4	3
17	0	6	3	-4	-3	2	6	7	-8	-10	4	6	4	4	-11	2	7	4	4	-7	0	8	10	-10
19	0	6	6	-6	-2	2	6	4	-5	-6	4	6	4	-4	-10	2	7	6	-7	-6	0	8	5	3
-23	1	6	7	6	0	2	6	3	-3	-1	4	6	5	4	-9	2	7	4	-3	-5	0	8	12	11
-22	1	6	6	5	1	2	6	4	-5	0	4	6	10	9	-8	2	7	4	-4	-4	0	8	3	-1
-14	1	6	5	4	2	2	6	4	-4	1	4	6	7	-6	-6	2	7	8	8	-3	0	8	5	5
-13	1	6	6	-7	5	2	6	16	15	3	4	6	5	5	-5	2	7	7	6	-2	0	8	4	-4
-11	1	6	3	2	6	2	6	10	9	5	4	6	7	7	-4	2	7	8	-8	2	0	8	4	4
-10	1	6	10	-10	7	2	6	3	-4	-18	1	7	5	5	7	2	7	16	-14	4	0	8	3	-2
-8	1	6	3	2	8	2	6	11	-11	-16	1	7	4	-4	8	2	7	6	3	5	0	8	4	5
-6	1	6	6	-6	10	2	6	5	-3	-15	1	7	7	-6	9	2	7	8	8	6	0	8	9	9
-5	1	6	4	4	12	2	6	4	-5	-14	1	7	4	-4	10	2	7	9	-9	7	0	8	24	-23
-4	1	6	7	-8	14	2	6	5	6	-12	1	7	3	-2	11	2	7	6	4	8	0	8	13	-13
-3	1	6	4	-4	15	2	6	9	-9	-11	1	7	5	-5	-12	3	7	8	-8	9	0	8	12	-11
-1	1	6	5	-5	16	2	6	6	4	-9	1	7	9	9	-11	3	7	5	-3	-14	1	8	4	-2
2	1	6	4	4	-13	3	6	9	-8	-6	1	7	7	7	-10	3	7	4	3	-13	1	8	11	-11
6	1	6	15	15	-11	3	6	11	11	-5	1	7	7	7	-9	3	7	17	18	-12	1	8	7	7
7	1	6	6	-6	-10	3	6	4	-1	-4	1	7	6	-6	-8	3	7	7	2	-11	1	8	6	6
8	1	6	3	-4	-8	3	6	6	6	-2	1	7	4	-5	-7	3	7	3	1	-10	1	8	5	3

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-8	1	8	9	-9	5	1	8	7	6	8	1	8	6	-5	-10	2	8	10	-9	-6	2	8	3	-2
-5	1	8	5	6	6	1	8	9	9	-11	2	8	10	-11	-9	2	8	5	3	2	2	8	11	12
-4	1	8	5	6	7	1	8	11	-11															

ANIL2

APPENDIX TEN

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	6	-7	-9	2	0	4	5	-11	4	0	3	-4	-5	-4	1	3	-2	0	-2	1	16	-15
3	0	0	3	3	-8	2	0	11	-11	-9	4	0	8	7	-4	-4	1	5	-5	3	-2	1	3	3
4	0	0	33	33	-6	2	0	8	-8	-8	4	0	4	-2	-2	-4	1	16	16	4	-2	1	5	-5
5	0	0	6	5	-5	2	0	19	-19	-7	4	0	3	5	-1	-4	1	14	-14	5	-2	1	5	5
6	0	0	49	49	-4	2	0	26	-26	-6	4	0	5	-5	5	-4	1	4	-4	6	-2	1	3	-2
7	0	0	15	-14	-3	2	0	25	25	-3	4	0	5	5	7	-4	1	7	-8	7	-2	1	5	5
9	0	0	5	-4	-1	2	0	17	-16	-2	4	0	3	3	8	-4	1	6	-6	10	-2	1	14	14
11	0	0	4	4	0	2	0	7	-6	-1	4	0	12	12	9	-4	1	10	10	11	-2	1	9	10
13	0	0	3	3	1	2	0	18	17	0	4	0	17	-17	10	-4	1	5	-4	12	-2	1	5	-5
-14	1	0	3	5	2	2	0	3	3	1	4	0	3	3	11	-4	1	4	-4	13	-2	1	5	4
-12	1	0	6	-6	3	2	0	36	-36	2	4	0	6	-6	-10	-3	1	8	7	-14	-1	1	4	-4
-10	1	0	22	23	4	2	0	5	5	3	4	0	3	4	-8	-3	1	3	-1	-12	-1	1	5	-5
-8	1	0	4	-3	7	2	0	3	-3	6	4	0	7	-8	-5	-3	1	7	7	-11	-1	1	12	13
-7	1	0	4	-3	9	2	0	20	-21	8	4	0	8	-8	-4	-3	1	6	6	-10	-1	1	13	-14
-6	1	0	22	22	10	2	0	10	10	-8	5	0	8	-9	-3	-3	1	12	-11	-9	-1	1	3	-3
-5	1	0	15	-15	12	2	0	5	4	-6	5	0	9	-9	-2	-3	1	7	-6	-8	-1	1	16	-15
-4	1	0	8	7	-13	3	0	6	5	-2	5	0	12	-12	-1	-3	1	8	9	-7	-1	1	16	-16
-3	1	0	28	-29	-11	3	0	7	7	-1	5	0	10	11	1	-3	1	6	-6	-6	-1	1	4	-4
-2	1	0	12	-12	-10	3	0	6	-7	0	5	0	13	-13	2	-3	1	6	-6	-5	-1	1	9	8
-1	1	0	31	-33	-9	3	0	17	17	1	5	0	5	6	3	-3	1	33	32	-4	-1	1	25	-24
0	1	0	9	-10	-7	3	0	4	4	4	5	0	3	-4	4	-3	1	6	-6	-3	-1	1	3	-4
1	1	0	25	-26	-5	3	0	11	10	5	5	0	4	5	6	-3	1	5	-6	-2	-1	1	35	-34
2	1	0	3	-4	-4	3	0	3	-3	-6	-5	1	5	-3	7	-3	1	3	-4	-1	-1	1	37	-36
3	1	0	30	-30	-3	3	0	15	14	-4	-5	1	3	2	9	-3	1	23	23	0	-1	1	64	64
4	1	0	8	-9	-1	3	0	3	3	-2	-5	1	9	9	10	-3	1	14	-14	1	-1	1	17	16
5	1	0	18	-18	0	3	0	3	3	0	-5	1	17	-17	11	-3	1	7	-6	2	-1	1	10	10
6	1	0	7	-7	2	3	0	13	13	2	-5	1	14	14	-12	-2	1	4	-4	3	-1	1	7	7
7	1	0	10	9	3	3	0	4	4	4	-5	1	3	3	-11	-2	1	11	11	4	-1	1	5	-4
8	1	0	8	7	4	3	0	7	6	6	-5	1	3	-2	-10	-2	1	3	-3	5	-1	1	3	4
9	1	0	20	-20	5	3	0	6	6	7	-5	1	4	2	-7	-2	1	17	17	6	-1	1	28	26
11	1	0	12	12	7	3	0	3	-2	8	-5	1	7	7	-5	-2	1	24	23	10	-1	1	7	-7
13	1	0	8	-8	8	3	0	14	14	-8	-4	1	10	11	-4	-2	1	9	-8	12	-1	1	8	-9
-11	2	0	9	-9	9	3	0	5	3	-7	-4	1	5	-5	-3	-2	1	9	9	13	-1	1	4	3
-10	2	0	18	-19	10	3	0	6	-7	-6	-4	1	5	-4	-2	-2	1	3	-3	-8	0	1	11	11

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-7	0	1	6	5	9	1	1	13	-13	0	3	1	8	8	-1	-5	2	10	-10	7	-3	2	16	-16
-6	0	1	9	9	10	1	1	11	10	1	3	1	9	8	0	-5	2	8	8	8	-3	2	7	7
-5	0	1	5	5	12	1	1	8	8	2	3	1	19	-18	1	-5	2	4	-4	9	-3	2	13	-13
-4	0	1	5	5	13	1	1	3	3	3	3	1	14	-13	3	-5	2	4	-3	11	-3	2	5	-5
1	0	1	18	-19	-14	2	1	4	4	5	3	1	6	-6	4	-5	2	8	9	-13	-2	2	6	6
2	0	1	26	-26	-13	2	1	7	-7	8	3	1	21	-22	5	-5	2	7	-8	-11	-2	2	4	4
3	0	1	19	-19	-12	2	1	7	-7	9	3	1	7	-6	6	-5	2	5	5	-10	-2	2	6	-6
4	0	1	24	-24	-11	2	1	10	10	10	3	1	3	4	-8	-4	2	4	4	-9	-2	2	8	8
5	0	1	15	-15	-10	2	1	6	6	-11	4	1	10	-11	-6	-4	2	4	3	-8	-2	2	8	-7
6	0	1	21	-20	-9	2	1	6	-6	-10	4	1	11	11	-4	-4	2	9	9	-7	-2	2	11	-11
7	0	1	7	-7	-6	2	1	6	6	-9	4	1	7	8	-2	-4	2	12	13	-6	-2	2	7	-7
8	0	1	7	-7	-5	2	1	11	11	-8	4	1	4	-5	-1	-4	2	14	-15	-5	-2	2	10	9
9	0	1	6	-5	-4	2	1	11	11	-6	4	1	4	-4	0	-4	2	4	4	-4	-2	2	18	-17
10	0	1	6	-6	-3	2	1	12	-11	-5	4	1	5	-4	2	-4	2	4	3	-3	-2	2	8	-8
-14	1	1	5	5	-2	2	1	13	13	-4	4	1	10	10	3	-4	2	4	4	-2	-2	2	14	-14
-12	1	1	14	14	-1	2	1	5	5	-3	4	1	5	6	4	-4	2	9	9	-1	-2	2	20	-20
-10	1	1	7	-7	0	2	1	13	13	-2	4	1	15	-16	5	-4	2	6	-6	0	-2	2	11	-11
-9	1	1	3	2	1	2	1	16	-16	-1	4	1	4	4	6	-4	2	6	6	1	-2	2	26	-24
-8	1	1	12	-12	2	2	1	18	17	0	4	1	5	-5	7	-4	2	4	-3	2	-2	2	23	21
-7	1	1	7	7	3	2	1	13	-13	1	4	1	3	3	8	-4	2	5	4	3	-2	2	3	3
-6	1	1	11	12	4	2	1	15	15	6	4	1	4	-5	9	-4	2	8	8	5	-2	2	3	-4
-5	1	1	10	-10	6	2	1	10	10	7	4	1	5	5	10	-4	2	4	3	7	-2	2	8	-8
-4	1	1	14	-15	8	2	1	9	9	8	4	1	10	10	-11	-3	2	5	-5	8	-2	2	28	28
-3	1	1	7	-7	9	2	1	8	-8	-4	5	1	8	-8	-10	-3	2	7	-7	9	-2	2	3	2
-2	1	1	38	-40	10	2	1	10	10	-3	5	1	7	8	-9	-3	2	5	5	10	-2	2	8	-8
-1	1	1	52	55	12	2	1	3	0	-2	5	1	16	16	-7	-3	2	3	-3	-13	-1	2	5	-4
0	1	1	32	33	-13	3	1	5	-5	-1	5	1	5	-5	-5	-3	2	5	-5	-12	-1	2	4	5
1	1	1	3	-3	-11	3	1	10	-10	0	5	1	9	-9	-4	-3	2	7	-7	-11	-1	2	11	11
2	1	1	15	15	-9	3	1	4	-5	1	5	1	6	5	-3	-3	2	13	13	-10	-1	2	10	-10
3	1	1	7	-6	-8	3	1	3	2	3	5	1	4	4	-1	-3	2	12	-12	-9	-1	2	5	-5
4	1	1	10	11	-7	3	1	4	-4	4	5	1	5	5	2	-3	2	3	3	-8	-1	2	12	12
5	1	1	17	17	-5	3	1	22	-21	5	5	1	5	-6	3	-3	2	14	-14	-7	-1	2	16	15
6	1	1	16	16	-4	3	1	8	8	-4	-5	2	3	0	5	-3	2	9	-8	-6	-1	2	15	14
8	1	1	12	12	-1	3	1	12	-12	-2	-5	2	11	11	6	-3	2	3	-3	-5	-1	2	30	29

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	-1	2	12	-12	9	0	2	7	7	-1	2	2	4	4	4	4	2	11	11	8	-3	3	25	25
-3	-1	2	14	14	11	0	2	9	-10	0	2	2	10	10	5	4	2	4	-4	9	-3	3	11	11
-2	-1	2	71	71	13	0	2	4	4	1	2	2	33	33	-7	5	2	3	-3	-13	-2	3	3	-4
-1	-1	2	53	53	-13	1	2	6	-5	2	2	2	10	-9	-4	5	2	10	10	-12	-2	3	3	2
0	-1	2	29	-29	-12	1	2	16	-16	3	2	2	3	3	-3	5	2	10	-10	-9	-2	3	13	-13
1	-1	2	29	29	-9	1	2	6	-6	4	2	2	3	-3	-2	5	2	9	8	-7	-2	3	8	-7
2	-1	2	16	16	-8	1	2	18	-18	6	2	2	9	9	-1	5	2	7	-7	-6	-2	3	7	6
3	-1	2	20	20	-6	1	2	19	-19	7	2	2	24	25	2	5	2	3	4	-5	-2	3	4	-3
4	-1	2	3	3	-5	1	2	19	19	8	2	2	25	-26	3	5	2	6	-6	-3	-2	3	23	-22
5	-1	2	12	12	-4	1	2	7	7	9	2	2	6	-7	-2	-5	3	4	4	-2	-2	3	27	26
6	-1	2	10	-10	-3	1	2	29	-30	-12	3	2	5	4	0	-5	3	7	-6	-1	-2	3	16	15
7	-1	2	6	5	-2	1	2	38	-39	-11	3	2	9	-9	2	-5	3	5	-6	0	-2	3	6	-6
8	-1	2	8	-7	-1	1	2	55	56	-10	3	2	7	7	5	-5	3	5	-5	1	-2	3	3	-3
9	-1	2	6	6	0	1	2	5	5	-8	3	2	4	5	6	-5	3	6	-6	2	-2	3	17	16
-13	0	2	4	-4	1	1	2	17	-16	-6	3	2	3	3	-7	-4	3	3	-1	4	-2	3	12	12
-12	0	2	17	18	2	1	2	9	-10	-5	3	2	10	-11	-5	-4	3	5	-5	5	-2	3	3	2
-11	0	2	10	10	4	1	2	8	-8	-4	3	2	11	11	-4	-4	3	14	-14	6	-2	3	10	-11
-10	0	2	13	-12	5	1	2	4	4	-3	3	2	8	8	-3	-4	3	18	18	7	-2	3	3	-1
-9	0	2	8	7	6	1	2	5	-6	-2	3	2	9	-9	-1	-4	3	10	-10	9	-2	3	5	-5
-8	0	2	13	-13	8	1	2	4	-3	-1	3	2	4	-4	0	-4	3	6	-6	11	-2	3	4	-4
-7	0	2	8	8	9	1	2	4	-4	0	3	2	3	-3	1	-4	3	9	10	12	-2	3	3	-2
-6	0	2	6	-7	11	1	2	6	6	1	3	2	3	3	9	-4	3	8	-7	-13	-1	3	10	-10
-5	0	2	7	8	-14	2	2	5	4	2	3	2	9	-10	-9	-3	3	9	9	-12	-1	3	11	11
-4	0	2	26	-27	-13	2	2	4	2	3	3	2	3	-3	-8	-3	3	4	3	-10	-1	3	5	4
-3	0	2	4	-3	-12	2	2	5	5	6	3	2	7	-7	-4	-3	3	3	4	-7	-1	3	7	-7
-2	0	2	106	-115	-10	2	2	9	9	8	3	2	4	3	-3	-3	3	11	11	-6	-1	3	16	16
-1	0	2	41	43	-9	2	2	7	-7	-11	4	2	6	-6	-1	-3	3	6	-6	-4	-1	3	4	5
2	0	2	27	-28	-8	2	2	3	4	-7	4	2	3	-4	0	-3	3	4	4	-2	-1	3	45	-46
3	0	2	5	-5	-7	2	2	16	15	-5	4	2	7	-6	1	-3	3	16	-16	-1	-1	3	14	-14
4	0	2	44	-44	-6	2	2	9	9	-4	4	2	5	-5	2	-3	3	11	10	0	-1	3	20	-20
5	0	2	37	35	-5	2	2	11	-10	-3	4	2	4	-4	3	-3	3	4	4	1	-1	3	9	-10
6	0	2	7	6	-4	2	2	9	9	-2	4	2	13	13	4	-3	3	5	6	2	-1	3	4	-5
7	0	2	6	-6	-3	2	2	3	-2	-1	4	2	7	-7	5	-3	3	3	2	3	-1	3	16	-16
8	0	2	7	-7	-2	2	2	7	7	0	4	2	6	7	7	-3	3	17	-17	4	-1	3	30	-30

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
5	-1	3	4	-5	1	1	3	17	18	6	3	3	21	22	2	-3	4	4	-4	-2	-1	4	27	25
7	-1	3	5	-5	3	1	3	5	-4	8	3	3	9	-9	3	-3	4	4	5	-1	-1	4	16	-16
9	-1	3	3	-3	5	1	3	9	9	-10	4	3	6	5	4	-3	4	3	-4	0	-1	4	10	-10
10	-1	3	5	6	7	1	3	17	17	-8	4	3	4	4	5	-3	4	7	7	1	-1	4	4	-3
11	-1	3	10	-11	8	1	3	8	-8	-6	4	3	9	-9	6	-3	4	5	-6	3	-1	4	3	3
-13	0	3	3	1	9	1	3	5	5	-4	4	3	21	22	7	-3	4	10	10	4	-1	4	22	21
-10	0	3	5	-5	10	1	3	6	-7	-3	4	3	4	-4	8	-3	4	7	-7	5	-1	4	11	-11
-9	0	3	3	-1	-13	2	3	4	-4	0	4	3	9	8	-11	-2	4	8	-8	7	-1	4	5	5
-8	0	3	3	-3	-9	2	3	7	-7	3	4	3	3	-4	-10	-2	4	9	9	8	-1	4	6	6
-6	0	3	3	-2	-8	2	3	4	-3	-8	5	3	4	3	-9	-2	4	7	8	9	-1	4	4	-4
-3	0	3	3	3	-7	2	3	4	-3	-5	5	3	5	-3	-8	-2	4	4	3	-14	0	4	16	-16
-2	0	3	6	6	-6	2	3	3	-4	1	5	3	4	-4	-6	-2	4	7	7	-12	0	4	12	12
-1	0	3	14	14	-5	2	3	8	8	-4	-5	4	5	-6	-5	-2	4	3	-2	-11	0	4	8	-8
0	0	3	18	18	-4	2	3	10	-11	-3	-5	4	7	6	-4	-2	4	26	26	-9	0	4	9	-8
1	0	3	9	9	-3	2	3	17	-17	-2	-5	4	5	-4	-3	-2	4	26	26	-8	0	4	6	-7
2	0	3	15	15	-2	2	3	3	-3	-1	-5	4	5	5	-2	-2	4	10	-9	-6	0	4	23	23
3	0	3	5	5	-1	2	3	18	18	0	-5	4	5	-5	-1	-2	4	7	7	-5	0	4	18	-18
4	0	3	11	12	0	2	3	22	-22	1	-5	4	4	4	0	-2	4	7	-6	-4	0	4	13	13
5	0	3	8	8	1	2	3	3	-2	3	-5	4	7	8	1	-2	4	6	6	-3	0	4	16	-16
6	0	3	8	8	2	2	3	17	-17	-6	-4	4	6	-6	2	-2	4	8	8	-2	0	4	15	16
8	0	3	5	5	3	2	3	4	-5	-4	-4	4	5	-4	3	-2	4	8	8	-1	0	4	24	-24
10	0	3	4	4	4	2	3	11	-10	-3	-4	4	12	12	5	-2	4	3	3	0	0	4	3	5
-14	1	3	9	-8	6	2	3	18	-19	-2	-4	4	3	-5	6	-2	4	20	-20	1	0	4	5	-6
-13	1	3	7	7	8	2	3	10	-9	1	-4	4	6	-6	7	-2	4	8	8	2	0	4	12	12
-12	1	3	11	11	-11	3	3	7	8	2	-4	4	6	-6	8	-2	4	9	9	3	0	4	24	-23
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-10	1	3	5	5	-7	3	3	5	4	4	-4	4	7	-7	-12	-1	4	4	3	7	0	4	5	-5
-9	1	3	3	-2	-5	3	3	5	5	6	-4	4	4	-3	-9	-1	4	4	-4	9	0	4	8	9
-7	1	3	15	15	-4	3	3	13	-13	7	-4	4	8	-7	-8	-1	4	4	-3	11	0	4	5	-6
-6	1	3	16	16	0	3	3	11	10	-6	-3	4	3	-2	-7	-1	4	6	-6	-12	1	4	5	5
-4	1	3	17	17	1	3	3	6	5	-5	-3	4	13	-12	-6	-1	4	7	7	-10	1	4	4	4
-3	1	3	24	-26	2	3	3	5	-6	-2	-3	4	9	-10	-5	-1	4	10	-10	-8	1	4	7	7
-1	1	3	14	15	3	3	3	4	4	0	-3	4	3	-3	-4	-1	4	27	-27	-7	1	4	9	-9
0	1	3	4	4	5	3	3	3	3	1	-3	4	6	5	-3	-1	4	9	-10	-6	1	4	3	4

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-5	1	4	12	12	0	3	4	8	8	3	-3	5	5	5	1	0	5	6	6	0	3	5	7	7
-4	1	4	12	12	2	3	4	4	5	5	-3	5	6	6	5	0	5	9	9	1	3	5	3	-3
-3	1	4	32	-32	6	3	4	7	7	6	-3	5	14	-14	9	0	5	3	1	2	3	5	5	-5
-2	1	4	9	9	7	3	4	5	-5	-6	-2	5	4	-3	-13	1	5	4	5	4	3	5	8	-8
-1	1	4	23	24	-7	4	4	4	3	-5	-2	5	4	4	-9	1	5	7	-7	6	3	5	4	4
0	1	4	6	5	-4	4	4	7	-7	-4	-2	5	16	-17	-8	1	5	5	-5	-6	4	5	8	-8
2	1	4	3	3	-3	4	4	3	4	-3	-2	5	11	-11	-7	1	5	6	6	-5	4	5	4	4
3	1	4	16	-15	-2	4	4	7	-7	-1	-2	5	4	-4	-6	1	5	6	-6	-2	4	5	5	-5
4	1	4	10	10	0	4	4	3	3	0	-2	5	13	-13	-5	1	5	7	7	1	4	5	4	3
5	1	4	13	13	1	4	4	5	-5	1	-2	5	3	-4	-4	1	5	7	-6	-5	-4	6	8	-8
6	1	4	8	-8	2	4	4	9	-9	2	-2	5	13	-14	-3	1	5	5	-5	1	-4	6	7	-7
7	1	4	10	-11	3	4	4	5	5	3	-2	5	3	-3	-2	1	5	6	-6	2	-4	6	4	5
-12	2	4	4	-2	4	4	4	4	-4	4	-2	5	10	11	-1	1	5	13	-13	-7	-3	6	3	4
-11	2	4	9	9	-6	5	4	5	-5	5	-2	5	7	-7	2	1	5	7	-7	-5	-3	6	5	6
-9	2	4	4	-4	-5	5	4	6	5	6	-2	5	12	-12	3	1	5	9	-8	-4	-3	6	5	5
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-4	2	4	13	-13	-6	-4	5	6	5	-6	-1	5	4	4	7	1	5	6	-6	5	-3	6	4	-4
-3	2	4	17	-18	-5	-4	5	7	-8	-4	-1	5	13	13	8	1	5	3	5	6	-3	6	8	8
-2	2	4	5	5	-4	-4	5	5	4	-3	-1	5	4	4	-13	2	5	5	5	-6	-2	6	16	-17
-1	2	4	7	-7	-3	-4	5	5	4	-2	-1	5	14	14	-10	2	5	3	-2	-5	-2	6	5	-6
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3	2	4	4	-4	2	-4	5	4	4	1	-1	5	10	10	-5	2	5	3	3	2	-2	6	11	12
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6	2	4	26	26	6	-4	5	5	5	4	-1	5	5	5	-2	2	5	13	12	-12	-1	6	4	-3
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-5	3	4	4	-3	-1	-3	5	3	3	-7	0	5	3	2	-4	3	5	10	-10	3	-1	6	9	9
-4	3	4	9	9	2	-3	5	4	4	-5	0	5	3	1	-3	3	5	5	3	5	-1	6	10	-10

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
6	-1	6	4	-4	1	1	6	8	8	-1	4	6	5	5	-3	0	7	3	-2	-5	3	7	4	-4
7	-1	6	7	7	2	1	6	7	-7	-7	-3	7	5	7	-2	0	7	3	-3	-1	3	7	3	3
-11	0	6	4	3	3	1	6	6	-6	-6	-3	7	12	-13	-1	0	7	5	-5	0	3	7	6	6
-9	0	6	5	-5	4	1	6	14	14	-5	-3	7	9	-11	0	0	7	3	-2	-6	-2	8	8	-8
-8	0	6	10	-10	5	1	6	11	11	-4	-3	7	7	7	3	0	7	7	-7	0	-2	8	8	-8
-7	0	6	13	13	6	1	6	4	-4	0	-3	7	7	-8	-9	1	7	4	-4	1	-2	8	4	3
-4	0	6	8	-8	-8	2	6	4	-3	1	-3	7	4	-4	-7	1	7	6	-6	-8	-1	8	4	4
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1	0	6	5	5	0	2	6	6	6	2	-2	7	5	-5	2	1	7	5	4	-5	0	8	8	-8
3	0	6	12	12	1	2	6	4	4	3	-2	7	4	4	3	1	7	9	8	-4	0	8	4	3
4	0	6	7	-6	2	2	6	7	-7	4	-2	7	5	4	4	1	7	6	-6	1	0	8	9	-9
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7	0	6	3	-4	6	2	6	4	-4	-10	-1	7	6	5	-9	2	7	3	-2	3	0	8	13	13
8	0	6	3	2	-10	3	6	3	-1	-4	-1	7	5	-5	-8	2	7	6	7	-8	1	8	6	5
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-5	1	6	4	4	0	3	6	4	-6	-2	-1	7	3	3	2	2	7	7	-7	-6	2	8	5	5
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-3	1	6	5	-5	-4	4	6	4	4	5	-1	7	8	-8										

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